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AGRICULTURAL RESEARCH ADMINISTRATION  
BUREAU OF AGRICULTURAL AND INDUSTRIAL CHEMISTRY

Report on Research Activity  
of the  
REGIONAL RESEARCH LABORATORIES  
in connection with  
the Eleventh Annual Meetings  
of the

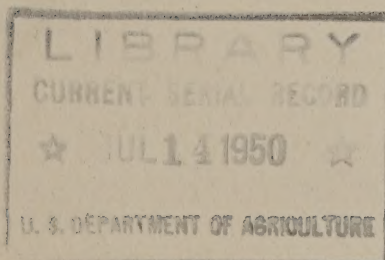
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<u>Region</u>	<u>Place</u>	<u>Dates</u>
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Washington  
June, 1950



REPORT OF THE  
COMMISSIONER OF THE  
BUREAU OF LANDS

REPORT ON THE  
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NEW YORK

REPORT OF THE  
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## CONTENTS

Page

SOUTHERN REGIONAL RESEARCH LABORATORY

COTTON UTILIZATION INVESTIGATIONS . . . . .	1
Cotton lint. . . . .	1
Fiber properties and reactions. . . . .	1
Properties of cotton cellulose. . . . .	2
Detection of honeydew on cotton . . . . .	3
Yarns spun from AHA cotton. . . . .	3
Improving the drafting step in processing . . . . .	3
New equipment for mechanical processing . . . . .	4
New cotton products by chemical modification. . . . .	4
Cottonseed . . . . .	5
Properties and reactions of gossypol. . . . .	5
Cottonseed storage. . . . .	6
Research on solvent extraction of cottonseed. . . . .	6
Cooperative work with commercial solvent-extraction plants. . . . .	7
Improving color and keeping qualities in cottonseed oil . . . . .	7
Fractionation and physical properties of monoglycerides . . . . .	7
Utilization of meal and protein . . . . .	8
SWEETPOTATO UTILIZATION INVESTIGATIONS. . . . .	8
Processing to recover starch. . . . .	8
Future research to emphasize food uses. . . . .	9
PEANUTS AND OTHER OILSEED CROPS UTILIZATION INVESTIGATIONS. . . . .	9
Solvent extraction of peanuts . . . . .	9
Production of peanut meal low in skin color . . . . .	10
Physical properties of fatty materials derived from peanut oil. . . . .	10
Effect of period of harvest of Texas-grown flaxseed . . . . .	11
Lipid content of rice bran. . . . .	11
Sesame oil. . . . .	11
Oil from <u>Cucurbita foetidissima</u> . . . . .	11
Safflowerseed oil . . . . .	12
RESEARCH AND MARKETING ACT. INVESTIGATIONS . . . . .	12
Cotton lint. . . . .	12
Fundamental characteristics of cotton fiber as a means of developing new uses . . . . .	12
Method of measuring the cross-sectional swelling of cotton fibers. . . . .	12
Oxidation of cotton cellulose. . . . .	12
Chemical bonding of cotton fiber with resins . . . . .	13
Reduction of crystallinity of cotton cellulose . . . . .	13
Applications of differential dyeing. . . . .	13
Development of new and improved products from cotton fiber through processing and chemical treatment. . . . .	13
Cotton in blends with other fibers . . . . .	13
Improving insect repellency of cotton bagging. . . . .	14
Effect of twist on weaving efficiency and quality of woven fabrics . . . . .	14
Contract research investigations . . . . .	14
Oilseeds and oils. . . . .	15
Improved methods of cottonseed oil extraction, new and improved prod- ucts from cottonseed . . . . .	15
Composition of cottonseed as influenced by environment and variety . . . . .	15
Fractionation of cottonseed meals. . . . .	15
Improved screw-processing of cottonseed. . . . .	15
Useful products from pigment glands. . . . .	16
Investigations of the nutritive value of cottonseed meals. . . . .	16



## SOUTHERN REGIONAL RESEARCH LABORATORY (Contd.)

## RESEARCH AND MARKETING ACT INVESTIGATIONS (Contd.)

Oilseeds and oils (Contd.)	
Quality improvement of peanut products and new uses for peanut oil. . .	17
Research aids peanut butter industry . . . . .	17
Uses for rice and rice byproducts and methods of processing . . . . .	17
Expanded research on fats and oils. . . . .	18
Research on monoglycerides . . . . .	18
Hydrogenation of fats and oils . . . . .	19

## WESTERN REGIONAL RESEARCH LABORATORY

ALFALFA UTILIZATION INVESTIGATIONS. . . . .	20
Growth-regulating factors . . . . .	20
Chemical constituents of alfalfa. . . . .	20
Research and Marketing Act projects. . . . .	21
Preservation of carotene in alfalfa meal. . . . .	21
FRUIT UTILIZATION INVESTIGATIONS. . . . .	21
Freezing preservation . . . . .	21
Dehydration . . . . .	22
Dehydrofreezing . . . . .	23
Canning and bottling. . . . .	23
Pectin. . . . .	23
Grape byproducts. . . . .	24
Torula yeast studies. . . . .	24
Vitamin B-12. . . . .	25
Research and Marketing Act projects. . . . .	25
New uses of deciduous fruits to prevent waste . . . . .	25
Spray drying studies on citrus fruits . . . . .	26
Continuous press for recovery of fruit juices . . . . .	26
Conversion of wastes into feedstuffs. . . . .	27
Fundamental studies of fruit tannins. . . . .	27
POULTRY UTILIZATION INVESTIGATIONS. . . . .	28
Freezing preservation of turkeys. . . . .	28
Egg proteins. . . . .	29
Frozen creamed chicken. . . . .	30
Calcined feathers . . . . .	30
Research and Marketing Act projects. . . . .	30
Precooked poultry and other dishes. . . . .	30
Preservation of dried egg . . . . .	32
VEGETABLE UTILIZATION INVESTIGATIONS. . . . .	32
Dehydration . . . . .	32
Freezing preservation . . . . .	32
Canning and other processing. . . . .	33
Hop constituents. . . . .	34
Freezing and cooling equipment. . . . .	34
Research and Marketing Act projects. . . . .	35
Uses for rice and byproducts, processing and handling . . . . .	35
Dried beans and peas. . . . .	35
Edible mushroom mycelium produced by submerged fermentation . . . . .	36
Conversion of potatoes to more stable forms and products. . . . .	36
Measurement of maturity in peas . . . . .	36
Sugar-beet processing liquors . . . . .	36



## WESTERN REGIONAL RESEARCH LABORATORY (Contd.)

WHEAT UTILIZATION INVESTIGATIONS. . . . .	37
Gluten phosphate. . . . .	37
Baking characteristics. . . . .	37
Milling characteristics. . . . .	38
WOOL UTILIZATION INVESTIGATIONS. . . . .	38
Research and Marketing Act Projects. . . . .	38
Fundamental studies to develop new and improved uses. . . . .	38
Methods of scouring wool. . . . .	39

## EASTERN REGIONAL RESEARCH LABORATORY

APPLE AND OTHER FRUIT UTILIZATION INVESTIGATIONS. . . . .	40
Juice products . . . . .	40
New portable essence unit . . . . .	40
Recovery of Concord grape essence during vacuum evaporation . . . . .	40
Market development. . . . .	41
Comparison of apple varieties for pie baking . . . . .	41
VEGETABLE UTILIZATION INVESTIGATIONS. . . . .	41
Vegetable wastes . . . . .	41
Processing studies. . . . .	41
Feeding trials. . . . .	42
Growing broccoli for feed . . . . .	43
Sapogenins for the preparation of anti-arthritis compounds . . . . .	43
TOBACCO UTILIZATION INVESTIGATIONS. . . . .	43
Tobacco essence and flavor. . . . .	43
Photochemical decomposition of nicotine. . . . .	45
MILK PRODUCTS UTILIZATION INVESTIGATIONS. . . . .	46
Protein investigations . . . . .	46
Bristle fiber from milk proteins. . . . .	46
Separation of caseins . . . . .	46
Carbohydrate investigations. . . . .	46
Recovery and purification of lactic acid. . . . .	46
Compounding and properties of Lactoprene EV . . . . .	47
ANIMAL FATS AND OILS UTILIZATION INVESTIGATIONS . . . . .	48
Basic composition of animal fats. . . . .	48
Stability and storage investigations. . . . .	48
Synthetic detergents from animal fats . . . . .	48
Unsaturated amides. . . . .	49
POTATO UTILIZATION INVESTIGATIONS . . . . .	49
Styrenated allyl starch . . . . .	49
Chemical and physical properties of potato starch. . . . .	49
Sloughing of potatoes . . . . .	49
Conversion of potatoes to stable form. . . . .	51
Drying of ground potatoes for flour and feed in a direct heat single pass rotary dryer. . . . .	51
TANNING MATERIALS, HIDES, SKINS, AND LEATHER INVESTIGATIONS . . . . .	51
Development of new and potential tanning materials . . . . .	51
Sumac . . . . .	51
Hemlock bark. . . . .	52
Saw palmetto. . . . .	52
Alum retannage of vegetable-tanned leather . . . . .	53



## EASTERN REGIONAL RESEARCH LABORATORY (Contd.)

INVESTIGATIONS CARRIED OUT UNDER THE RESEARCH AND MARKETING ACT . . . . .	53
Apiary products. . . . .	53
Dried honey - skim milk . . . . .	53
Flavor removal. . . . .	54
Crystallized honey - fruit spread . . . . .	54
Disposal of dairy wastes . . . . .	54
New and improved uses of deciduous fruits especially to prevent waste. .	54
Biochemistry of sour cherries . . . . .	54
Investigations of basic properties and functions of tannins and develop-	
ment of new tanning materials . . . . .	55
Production of canaigre. . . . .	55
Processing studies. . . . .	56
Processing <i>Nicotiana rustica</i> as a source of nicotine . . . . .	56
Improvement of quality and flavor of maple sirup and other maple products	57
High-flavored maple sirup . . . . .	57
Permanent color standards . . . . .	57
Conversion of potatoes to more stable form and products; new methods of	
using potatoes for feeds and non-food uses. . . . .	58
Conversion of surplus potatoes to stable form . . . . .	58
Natural freeze drying of potatoes . . . . .	58
Utilization of wool grease and other wool byproducts . . . . .	59
Conversion of vegetable and animal byproducts, surpluses and wastes into	
new feedstuffs and the establishment of their feeding values. . . . .	59
Seabrook Farming Corporation contract . . . . .	59
Processing field and cannery wastes . . . . .	60
Production of amino acids from milk proteins . . . . .	60
Expanded research on fats and oils . . . . .	60

## NORTHERN REGIONAL RESEARCH LABORATORY

AGRICULTURAL RESIDUES UTILIZATION INVESTIGATIONS. . . . .	62
Yield of pentose sugar from corncobs increased. . . . .	62
Fermentation of pentosan hydrolyzate liquors. . . . .	62
Composition of corncobs . . . . .	62
Hydrogenation and dehydrogenation products from furfural. . . . .	63
Structure and synthesis of gymnosperm lignin. . . . .	63
Mechano-chemical process developed for pulping crop residues. . . . .	63
9-Point corrugating board improved. . . . .	64
Fine straw pulp . . . . .	64
Soft-grit blasting. . . . .	64
CORN, WHEAT, AND OTHER CEREAL CROPS UTILIZATION INVESTIGATIONS. . . . .	65
Fundamental studies on starch granule structure . . . . .	65
Starch sponge as a hemostatic agent . . . . .	65
Periodate oxidation of carbohydrates. . . . .	65
Improved techniques for the study of amylose hydrolysis by malt	
alpha-amylase. . . . .	66
Synthesis of d-glucuronic acid. . . . .	66
Synthetic detergents derived from dextrose. . . . .	67
Production of potassium acid saccharate . . . . .	67
Production and utilization of fungal amylase. . . . .	68
Production of citric acid from glucose. . . . .	68
Production of sodium gluconate. . . . .	69



## NORTHERN REGIONAL RESEARCH LABORATORY (Contd.)

CORN, WHEAT, AND OTHER CEREAL CROPS UTILIZATION INVESTIGATIONS (Contd.)	
Polymyxin - an antibiotic . . . . .	69
Manual of the <u>Penicillia</u> . . . . .	70
Cyanoethylation of alpha-amino acids. . . . .	70
Study of alcohol for motor fuels in automotive engines. . . . .	70
SOYBEANS AND OTHER OILSEED CROPS UTILIZATION INVESTIGATIONS . . . . .	71
Contributing causes to flavor instability of soybean oil. . . . .	71
Commercial soybean oils . . . . .	71
Flavor stability of oils other than soybean . . . . .	72
Characteristics of esters and acids . . . . .	72
Refining loss and sludge in soybean oil . . . . .	72
New method of locating the double bond in isomers of oleic acid . . . .	73
Cooperative work on improvement of nutritional value of soybean oil meal . . . . .	73
Pilot-plant production of gelable soybean protein - Gelsoy. . . . .	73
RESEARCH AND MARKETING ACT PROJECTS . . . . .	74
New structural and chemical studies of grain kernels. . . . .	74
Production of riboflavin by <u>Ashbya gossypii</u> . . . . .	74
Vitamin B-12 synthesis by microorganisms. . . . .	75
Fungal amylase process for plant-scale conversion of sound grains into alcohol . . . . .	75
Alcohol-water injection studies on internal combustion engines. . . .	75
Linolenic acid and the flavor stability of soybean oil. . . . .	76
Isolation and characterization of flavor principles . . . . .	76
Composition of soybean phosphatides . . . . .	76

## AGRICULTURAL CHEMICAL RESEARCH DIVISION

SOUTHERN FRUITS AND VEGETABLES PROCESSING INVESTIGATIONS. . . . .	77
Investigations of Texas and Florida citrus. . . . .	77
New knowledge of enzymes an aid to the pickling industry. . . . .	78
CANE SUGAR, SIRUP, AND BYPRODUCTS INVESTIGATIONS. . . . .	78
Clarification of cane juice and recovery of acetic acid studied . .	78
Research for the candy industry . . . . .	79
TUNG NUTS INVESTIGATIONS. . . . .	79
Tung press cake dust an explosive hazard. . . . .	79
New methods of analysis provided tung industry. . . . .	79
RESEARCH AND MARKETING ACT INVESTIGATIONS . . . . .	80
New and improved uses of citrus products. . . . .	80

## DIVISION OF BIOLOGICALLY ACTIVE COMPOUNDS

BASIC INVESTIGATIONS IN THE CHEMISTRY OF AGRICULTURAL PRODUCTS. . . . .	81
Plant disease. . . . .	81
Further characterization of tomatine and tomatidine . . . . .	81
Plant-growth regulators. . . . .	81
Radioactivity aids growth-regulator research. . . . .	81
New synthetic compounds tested for growth-regulating properties . . .	82
INVESTIGATIONS CARRIED OUT UNDER THE RESEARCH AND MARKETING ACT . . . . .	82
Production of plant-growth regulating compounds from agricultural sources	82
Corn pollen and distillers solubles . . . . .	82



## DIVISION OF BIOLOGICALLY ACTIVE COMPOUNDS (Contd.)

## INVESTIGATIONS CARRIED OUT UNDER THE RESEARCH AND MARKETING ACT (Contd.)

Production of antibiotics from agricultural sources. . . . .	83
Survey of various plants and plant parts as sources of antibiotics. .	83
Antibiotics from the sweetpotato plant. . . . .	83
Antibiotics from cabbage. . . . .	83
Antibiotics from bananas. . . . .	83

## ENZYME RESEARCH DIVISION

Work on citrus. . . . .	85
Work on cereals. . . . .	85
RESEARCH AND MARKETING ACT PROJECT. . . . .	86
New and improved uses of citrus products. . . . .	86

## LABORATORY OF FRUIT AND VEGETABLE CHEMISTRY

Fruit products. . . . .	87
Vegetable products. . . . .	87
RESEARCH AND MARKETING ACT PROJECTS . . . . .	87
New laboratory at Pasadena, Calif. . . . .	87
New and improved uses of citrus products. . . . .	88

## FRUIT AND VEGETABLE PRODUCTS LABORATORY

Fruit products. . . . .	89
Vegetable products. . . . .	89
RESEARCH AND MARKETING ACT PROJECT. . . . .	89
Recovery of edible juice from pear waste. . . . .	89

## MICROBIOLOGY RESEARCH DIVISION

MICROBIOLOGY OF DRIED EGGS AND RELATED EGG PRODUCTS . . . . .	90
Development of an improved method for the fermentation of egg white .	90
The antibiotic activity of polymyxin and other anti-bacterial agents	
against <u>Salmonella</u> organisms isolated from dried egg and related egg	
products . . . . .	90
PREPARATION OF PRE-COOKED FROZEN FOODS, AND THE POSSIBLE OCCURRENCE AND	
SIGNIFICANCE OF PATHOGENIC ORGANISMS IN FROZEN FOODS . . . . .	91
Occurrence and significance of <u>Salmonella</u> organisms in commercially	
frozen foods . . . . .	91
IMPROVEMENT OF MICROBIAL QUALITY OF EGGS, EGG PRODUCTS, POULTRY, AND	
POULTRY PRODUCTS . . . . .	91
Improvement of microbiological methods for the detection of <u>Salmonella</u>	
organisms in dried eggs and related egg products . . . . .	91
Investigation of the pathogenicity for humans of <u>Salmonella</u> organisms	
isolated from dried egg and related egg products . . . . .	92



## NAVAL STORES RESEARCH DIVISION

NAVAL STORES PRODUCTION, PROCESSES, AND EQUIPMENT INVESTIGATIONS. . . . .	93
Selection of cups for acid-stimulated gum . . . . .	93
Improved processing methods and equipment . . . . .	93
Help when needed. . . . .	93
INVESTIGATIONS OF THE COMPOSITION, PROPERTIES, COMPONENTS AND DERIVATIVES OF NAVAL STORES. . . . .	94
Composition of acid-stimulated pine gum . . . . .	94
New compounds from turpentine . . . . .	94
INVESTIGATION OF USES, HANDLING, AND TRANSPORTATION OF NAVAL STORES . . . .	94
New type of metal resinate developed. . . . .	94
RESEARCH AND MARKETING ACT INVESTIGATIONS . . . . .	95
Pilot-plant production of maleo-pimaric acid from gum rosin . . . . .	95

## PHARMACOLOGY LABORATORY

PHARMACOLOGICAL INVESTIGATIONS. . . . .	96
---	----

## NATURAL RUBBER EXTRACTION AND PROCESSING INVESTIGATIONS

Milling of lush shrub . . . . .	98
Deresination. . . . .	98
Resin characterization. . . . .	98
Low-temperature testing of guayule rubber . . . . .	99
Aging studies . . . . .	99







COTTON UTILIZATION INVESTIGATIONS

COTTON LINT

Fiber Properties and Reactions

Thermodynamic investigations of the relations between water and the cellulose of cotton, differentiating between the water in the cotton that will freeze and that which will not freeze, have shown the treatments of the cotton which result in an increase in the nonfreezing water content are those which lead to (1) greater penetration of dyes and finishing agents; (2) higher moisture absorption; and (3) in general, enhancement of those properties of reactivity which are associated with a greater available surface.

The protein nature of the lumen contents of the cotton fiber has been definitely established and several of the constituent amino acids have been identified. This fundamental information will be of considerable value in purification processes.

The average of and range in the amounts of the traces of copper, iron, and manganese that may occur in virgin cotton fiber and in cottonseed kernels have been determined by spectrochemical techniques. Knowledge of these traces of metals is considered of value in relation to degradation during textile finishing.

Fiber length-strength investigations established the variability in breaking load and elongation-at-break of individual fibers within a sample. In general, the relation between fiber length and a particular physical property was similar for most cottons tested. A proposed strength index, determined from tests on fibers of three modal length groups, had a linear relation with the average fiber strength of all fibers in a sample. The sampling method used to determine the strength index reduces the number of fibers required for a test. Its use will simplify sampling procedures when a comparison of chemically treated cottons with raw cottons must be made on the basis of properties of individual fibers.

While the precision of the flat-bundle method for determination of extent of mechanical damage to cotton fibers is insufficient for any critical analysis of changes in strength, there was no indication of extensive fiber damage from mechanical processing. These results were confirmed by strengths of individual fibers taken from the cottons and the fluidity values of samples as determined by our cuprammonium fluidity method. The stress-strain



analysis of processed cotton fibers gave a reduced elongation-at-break and a difference in the shape of the stress-strain curves. The change in strength was probably within experimental error. A decrease in elongation-at-break can be attributed to removal of kinks and secondary creep within the fiber as stresses are applied during the several mechanical processes.

### Properties of Cotton Cellulose

Fractionation studies have shown the possibility of isolating cellulose samples of homogeneous composition, modified only by possessing one glycosidic methoxyl group per molecule. Hitherto, success in this field has been achieved only with the highly modified cellulose derivatives, such as soluble acetates and nitrates. Such material offers the possibility of studying the effect of chain length distribution on the physical properties of cellulose.

Empirical relations between intrinsic and specific viscosity of cellulose dispersion in cuprammonium and cupriethylenediamine solutions will allow intrinsic viscosity values to be obtained by means of a single viscosity measurement at a specified concentration instead of the 4 or more measurements hitherto required. Failure to find evidence of decrease in degree of polymerization in cotton cellulose as a result of mechanical processing, as reported in recent literature, leaves this important question unsettled, but indicates that such degradation is probably not nearly so severe as the recent literature reports indicate -- if, indeed, it occurs at all.

A difference in fine structure of mature and immature cotton fibers has been demonstrated by means of the x-ray spectrometer employing the newly developed rotating specimen mount. The fibers were separated after being dyed according to the differential dye test technique and the degree of orientation measured. Results have indicated a significant difference in the degree of orientation of mature and immature fibers. The mature were found to be more highly oriented.

The so-called limiting degree of polymerization (D. P.) is believed to indicate the approximate length of the crystallites. Since, on the basis of the findings and hypotheses of other workers, it was conceivable that the condition of hydrolysis (temperature, acid concentration) might produce "limit hydrocelluloses" of different degrees of polymerization, the effects of these variables on the limiting D.P. have been explored in considerable detail. The uniform relation between weight loss and D.P. which has been found will serve as an extremely useful guide to the extent of hydrolysis necessary, in studying future samples, to produce limit hydrocellulose and this will help evaluate cotton for the types of usage for which it is suited.



### Detection of Honeydew on Cotton

A test for the detection of honeydew, the excreta of aphids, has been selected and offered for use to the cotton industry for the examination of raw cotton for honeydew contamination. The presence of honeydew in an appreciable amount causes cotton to be sticky and troublesome in opening and cleaning in the textile mill. The prominence given the report on the test in the trade journals and the requests for copies of the publication show the strong interest of the cotton industry in the test offered.

### Yarns Spun from AHA Cotton

Yarns spun from the strong-fibered AHA 6-1-4 variety (staple length 1-1/8 inches) were as strong as those spun from a long, fine-fibered cotton whose staple length was 1-1/2 inches. This observation is significant since the AHA cotton is fully 3/8-inch shorter than the longer variety. It has also been shown that when this strong-fibered cotton is considered in combination with a weak-fibered variety (both cottons being of equal staple length), yarns spun from the two differed in elongation, with the AHA having the lower.

### Improving the Drafting Step in Processing

Continuation and expansion of draft studies are justified on the grounds of their practical application in improving the quality of cotton products and at the same time lowering processing costs. About 100 requests have been received for reprints of the publication of the previous studies and for other information. These inquiries originated with spinners (domestic and foreign) and textile schools. There was expressed demand from industry that the draft proportionment studies be enlarged to include short- and medium-staple length cottons as well as other drafting systems commonly in use by industry.

Contrary to the recommendation by the machinery manufacturers that 100 feet per minute is approximately the best speed for quality yarns, our studies indicated that higher speeds do not adversely affect the quality. Executives of 15 mills visited recently expressed the opinion that existing textile processing machines are capable of producing products of higher quality than is generally being obtained. They expressed the need for more technical data than are currently available. Data from studies thus far undertaken point to possibilities of obtaining these higher speeds and lower processing costs at the drawing process without sacrificing yarn quality.



### New Equipment for Mechanical Processing

The gas-fired infrared slasher, complete with automatic controls, constitutes a progressive step in designing textile equipment on modern scientific principles. Its performance has interested the textile mills, but commercial adoption depends upon a more complete determination of the ability of the slasher to meet production standards in everyday operation.

The Southern Laboratory cotton opener machine is an important contribution to the processing of mechanically harvested and other trashy cottons. It puts lint cotton in an optimum condition for cleaning by existing textile equipment, thereby overcoming one of the current obstacles hindering the expansion of farm mechanization methods. The value of the new opener is recognized and indications are now that it will be adopted by the textile industry.

The attachment developed by Southern Laboratory engineers for weaving high-pickage fabrics on conventional textile looms is proving mechanically sound for industrial utilization. Fabrics with 38 percent more filling threads per inch than normal have been successfully woven. It is believed the attachment may increase cotton utilization through the development of very dense water-repellent fabrics and specialty fabrics for industrial use. These close-woven goods should make cotton more competitive with synthetic fibers for outer garment apparel, tentage, tarpaulins, and in other fields where water and air resistance are factors.

### New Cotton Products by Chemical Modification

The new combined yarn and cloth processing machine with all the units in a continuous range, and the continuous partial acetylation process being developed by its aid, promise the replacement of slow expensive batch methods, heretofore employed, by a much more rapid and less expensive process for preparing partially acetylated cotton. This is a definite step toward commercial production of partially acetylated and other modified cottons for uses where other textiles fail upon exposure to high temperatures on long heating; or where they mildew and rot.

Work on a number of cottons of very different properties, in yarn form, indicates that simple mercerization techniques can be employed to help characterize the cottons, as well as to show how yarns and fabrics made from them are likely to respond to commercial mercerizing. For example, it has been clearly shown that mature cotton responds better than immature to mercerization; also that some irrigated cottons respond better than others.



The development of a soluble cotton yarn by the carboxymethylation treatment is of considerable importance to industry as evidenced by approximately 50 requests for samples of the yarn and by the fact that three industrial concerns have sent representatives to this laboratory to discuss properties and applications of the soluble yarn.

New advantages have been shown in the resistance of insoluble carboxymethylated cloth to soiling, and crease-proofing treatments can be applied to the cloth without the use of a catalyst with superior results. License to use the patent for producing the partially carboxymethylated cloth has been requested and pilot-plant batches have been processed by an industrial finishing company using commercial mercerizing equipment.

Aminized cotton, which has been prepared in fabric form on pilot-plant scale, has been found to possess several new and interesting properties. It has an increased affinity for direct cotton dyes; and in addition has considerable affinity for acid wool dyes which do not react with unmodified cotton. It serves as an anion-exchange material, and means have been found to produce a treated fabric of this type with an exchange capacity comparable to the commercial anion-exchange resins. This fabric may find specialized applications where an anion-exchange material in fabric form is advantageous. Because of its increased chemical activity, the aminized cotton offers a promising base material for additional modification of cotton.

It has been further demonstrated that cotton can be so chemically modified as to have acid and base ion-exchange strengths comparable to commercial ion-exchange resins. Judging from inquiries there is a considerable interest in the potential use of ion-exchanges in the form of a fabric.

## COTTONSEED

### Properties and Reactions of Gossypol

Isolation of gossypol (the yellow pigment of cottonseed) from a chloroform solution of gossypurpurin (the purple pigment of cottonseed), which had been treated with concentrated hydrochloric acid, has provided information on the relationship of these two pigments which are present in cottonseed. Additional information concerning the reactions of these two pigments is helping in an understanding and solution of the problems involved in bleaching and color reversion of cottonseed oil during and following commercial processing.

Methods have been developed for the determination of total gossypol pigments in cottonseed meals and oils which are expected to serve as useful evaluation tools in research on cottonseed processing



and on improving the nutritive value of cottonseed meal. On the basis of the present use of the method previously published for the determination of free gossypol pigments by the cottonseed industry, these more recently developed procedures will be used extensively for both plant control and research in the industry.

### Cottonseed Storage

A mixture of relatively cheap and nontoxic chemicals has been found to inhibit deterioration in cottonseed during storage equally as well as more expensive mixtures previously used. These compounds, acetic acid and sodium metabisulfite, should make possible low cost treatment of seeds to prevent deterioration. Extensive laboratory storage tests are being conducted on this mixture.

Pilot-plant storage tests in cooperation with the Mississippi Agricultural Experiment Station have indicated that chemical treatment does improve the storage properties of planting seed. Seedlings treated with chemicals prior to storage exhibited more rapid growth than did untreated seedlings stored the same length of time. These results have encouraged the research workers at the State Experiment Station to conduct a second series of storage experiments on a more extensive scale.

### Research on Solvent Extraction of Cottonseed

An investigation of the effect of seed moisture (4-14 percent) upon hulling, purifying, cracking and flaking and on the flakes produced showed that the control of moisture is not only of extreme importance for the preparation of flakes the most suitable for direct solvent extraction, but also has a direct bearing on many of the operations in the extraction process. The optimum conditions for preparation of flakes have been established. This information is extremely valuable in pilot-plant work of this Laboratory and to industrial processors for determining the best conditions for flake preparation, and also to predict the problems which may be encountered in the solvent extraction of flakes from cottonseed at various moisture levels.

Equipment modifications and improvements in the operation of the continuous solvent-extraction pilot plant and also the production of a most suitable flake material were together responsible for the successful continuous solvent extraction of cottonseed with commercial hexane at 150 pounds per hour. The residual oil content in the meal was less than 1 percent. The miscella contained less than 0.4 percent "fines" by weight of the feed flakes. The fines in the miscella was only 10 to 15 percent of the amount said to be produced in commercial solvent-extraction installations of several different designs. These results for direct solvent extraction indicate that cottonseed flakes prepared under optimum conditions can be processed



more efficiently than is presently realized by most of the solvent plants.

### Cooperative Work with Commercial Solvent-Extraction Plants

Working conferences have been held with managers and superintendents of cooperative cottonseed oil mills. Analysis and evaluation of their operations and products have been made, tabulated, and discussed with members of each mill. Technical assistance was given, and recommendations were made toward the solution of some of their immediate problems. The cooperative work has proved valuable to the Laboratory not only in that a direct insight of the operations at these mills has been obtained but also it has made evident the direction in which this Laboratory should perform some of its research on cottonseed if direct extraction of cottonseed flakes is to be established as a feasible and economical process.

### Improving Color and Keeping Qualities in Cottonseed Oil

Results obtained in the investigation of countercurrent and batch bleaching of cottonseed oil in solvents and regeneration of the spent bleaching clay indicate that such methods are highly efficient and economical with respect to the amount of bleaching clay required to effect a given reduction in color, and because the spent clay can be regenerated and reused many times without substantial loss in bleaching power. Even though cottonseed oil is relatively resistant toward rancidification (development of undesirable odors and flavors) compared to many other edible oils it is nevertheless desirable to extend this natural resistance toward deterioration to a maximum. An increase of 9-fold in the time required for cottonseed oil to develop rancidity under accelerated tests was obtained by the addition of extremely small concentrations of unhardened and hardened cottonseed oils.

### Fractionation and Physical Properties of Monoglycerides

Technical-grade monoglycerides, as presently manufactured from vegetable oils, are a mixture of free glycerol, monoglycerides, diglycerides, and triglycerides in which the actual content of monoglycerides usually ranges between 30 and 60 percent. The work on purification of the technical products has shown that they can be purified by relatively simple procedures and the proportion of the desired monoglyceride can be doubled and tripled. For many present uses and for other potential uses, the purified monoglycerides are superior to the original technical-grade product. This is especially true in their use for the preparation of a variety of new compounds which have heretofore not been developed extensively because of the lack of purity of the commercial products that have been available.



While monoglycerides are used extensively as emulsifiers and surface-active agents, little is known about their surface-active properties, principally because the compounds have attained industrial utility only within the past 10 or 15 years. Quantitative knowledge with respect to the relative ability of various monoglycerides to lower the interfacial tension at oil-water interfaces will have a marked effect in extending the uses for these compounds in shortening, margarine, cosmetic preparations, and similar products.

#### Utilization of Meal and Protein

Solvent-extracted cottonseed meal was used as the glue in manufacture of plywood. Plywood bonds thus made had sheer strength slightly lower than those made with commercially-prepared casein glues. The wet strength of the joints prepared with cottonseed meal glue was superior to joints prepared with commercial protein glues. This improved wet strength may serve as the basis for the use of cottonseed meal in the plywood industry.

Fiber has been produced from cottonseed protein dispersions. The fact that these fibers have a dry strength of 74 grams per denier and a wet strength of 0.25 grams per denier indicates that industrially useful textile fiber can be made from cottonseed protein. In a preliminary experiment, in cooperation with an industrial firm, cottonseed protein was successfully dry-extruded in a manner similar to the production of curled casein monofilament. This is probably the first time that cottonseed protein has been extruded to form a filament in this manner and indicates possibilities for future development along these lines.

Pilot-plant work on preparation of protein from cottonseed meal has made evident that further investigations must be made to find a method for producing light-colored cottonseed protein of high nitrogen content without substantially reducing its yield. If cottonseed protein is to be utilized for industrial uses, such as fibers, adhesives, sizes, and coatings, then pilot-plant work, supported by laboratory findings, for improving the quality and color of the protein should be continued.

### SWEETPOTATO UTILIZATION INVESTIGATIONS

#### Processing to Recover Starch

With improved and simplified processes for extraction and refining of sweetpotato starch and for recovery of byproduct feed protein developed and proved on a large pilot-plant scale, investigations relating to the manufacture and utilization of sweetpotato starch have been carried as far as justifiable until the economy of farm production of sweetpotatoes so changes as to permit profitable growing of the crop for industrial use. Research during the past year



indicated that the use of a saw-blade rasp as a first grinder, followed by a screen to remove coarse pulp fragments of low starch content, reduces the cost and power requirement of the first grinder and makes it easier to eliminate fine fiber in the finished starch.

#### Future Research to Emphasize Food Uses

Continued investigations will place major emphasis on the utilization of the considerable proportion of the tablestock sweet-potato crop which consists of No. 2's and sound jumbos and which to an increasing extent is difficult or impossible to move in the fresh market, with the rapidly increasing trend toward rigorous grading and restriction of the market to No. 1's or better. Improved processed food products, especially canned sweetpotatoes, offer the most tangible prospect as an outlet if some problems of processing can be solved. Solution of the problem of excessive softening in processing of small whole sweetpotatoes or cut pieces often encountered with fresh-dug roots and almost universal with material cured and stored for any length of time, and development of a solid or mash pack of superior flavor and consistency, namely, one like a prime baked sweetpotato, would permit canning of all No. 2 and sound jumbo sweetpotatoes. Another prospective outlet is frozen puree for specialty food products and home use.

#### PEANUTS AND OTHER OILSEED CROPS UTILIZATION INVESTIGATIONS

##### Solvent Extraction of Peanuts

Up to now the commercial application of continuous solvent extraction to peanuts has met with negligible success because of the inherent property of peanut flakes to disintegrate into a virtual powder during extraction. Nevertheless, because of the relatively high cost of producing peanuts on the farm, it may be necessary to recover the oil by the solvent process in order to obtain added values in the products -- a higher recovery of oil and a meal of greater utility for food and industrial use.

Significant progress has been made in establishing optimum processing conditions for the successful solvent extraction of peanuts. Two pilot-plant runs were completed after essential modifications had been made to improve the mechanical operation of the extractor. These modifications previously had resulted in the highly successful extraction of cottonseed flakes to a low lipids content. In applying this know-how to the processing of peanuts, exceptionally smooth operation of the plant was obtained for the first time in a run using normally prepared flakes. In another run, where special heat-treated flakes were used, the overall operation was the best ever attained. With this information at hand, it is reasonable to expect that the residual oil content of



the meal can be reduced to 1 percent through a combination of proper flake thickness and careful control of the heat-treating operation.

#### Production of Peanut Meal Low in Skin Color

The lye and the water treatment processes developed by the Southern Laboratory are the only effective methods known for the removal of the objectionable skin color from U. S. No. 1 grade peanuts. The use of such a treatment prior to solvent extraction is a requisite if the defatted meal is to serve as a source of protein for large-scale industrial utilization.

During the year, around 8,000 pounds of peanuts were lye-treated for the removal of skin color. The production of this material, along with several hundred pounds of peanuts treated by the water process, made possible the preparation of a stock pile of several thousand pounds of defatted meal, low in skin color. Wherever possible, in the course of preparing this material, efforts were made to develop improved techniques and to obtain data which will be useful in placing both processes on a continuous pilot plant basis.

It has been demonstrated that the color of the red skins of peanuts is largely due to the presence of approximately 7 percent of a catechol tannin of high molecular weight. This information is important in the further development of methods for the production and evaluation of peanut protein products free of objectionable color.

#### Physical Properties of Fatty Materials Derived from Peanut Oil

Investigations of the solubility effect in mixtures of fatty acids with solvents have made it possible to predict to what extent separation of the acids into individual components can be effected. This information is of direct use in the separation of mixtures by any of the commercial fractional crystallization processes, which have assumed considerable importance within the past few years.

Measurements of the heat characteristics of hydrogenated and unhydrogenated peanut oils and products derived from them, and of the changes which occur in these materials under different conditions of cooling and processing, have given results intimately related to practical problems concerning the consistency, or "body," of plastic fatty products intended for edible purposes. From the consumer's viewpoint, that quality known as "consistency" is highly important in many types of fat products, particularly shortening, margarine, butter, lard, and the like.

Since fatty materials are able to exist in more than one crystal form, knowledge of these different forms, and of the conditions whereby they are able to interchange, is of value in efforts to impart desirable characteristics to finished fatty products.



### Effect of Period of Harvest of Texas-Grown Flaxseed

Information is now available on the changes in flaxseed grown in Texas throughout the harvest period. Deterioration is greater in flax harvested late in the season, and the tendency for further deterioration is increased as the season advances. This information is valuable to processors and growers because it emphasizes that flaxseed is not a uniform material throughout the harvest season. Hence, the grower will be more careful in selecting optimum harvest conditions and the processor will be able to adjust his conditions of storage and processing to minimize the more rapid deterioration that takes place later in the season.

### Lipid Content of Rice Bran

Systematic analyses of rough rice of 8 varieties from productions at 3 locations in 1948 have shown the true pericarp and germ fraction (bran) to contain an average of 21.79 percent of lipids with individual values varying from 18.41 to 23.35 percent.

### Sesame Oil

The characteristics and composition of sesame oil of domestic origin have been determined, and methods have been developed for quantitative determination of the minor constituents -- sesamol, sesamolin, and sesamin. The relation of these components to the stability of the oil has been established. The method developed for the determination of sesamin in sesame oil is more specific for this component and is simpler to carry out than is the existing method used commercially. The new method should prove useful in selecting sesame oils of high sesamin content for use as synergists in pyrethrum insecticides, since sesamin is the only component of sesame oil which has been definitely proven to possess synergistic activity.

The information concerning sesame oil which has been obtained under this project should be valuable to processors of sesame seed and oil in producing an oil of high stability through control of processing conditions to retain high antioxidant (sesamol) content. One processor of sesame oil who produces a sesamin extract for use in insecticides, has reported that these investigations have furnished information of great value to him as well as to consumers of the desaminized oil in margarine.

### Oil from Cucurbita Foetidissima

Oil from the seed of the Buffalo Gourd, C. foetidissima, has been characterized and found to be suitable for use as a salad or cooking oil, but it has a somewhat higher color and lower stability than other vegetable oils. The hydrogenated oil did not have as light a color as is desired for high quality commercial shortenings.



### Safflowerseed Oil

Safflowerseed oil of domestic origin has been found to have limited utility in edible products. Salad or cooking oil produced from safflowerseed undergoes an undesirable flavor change similar, but not identical, to reversion. For good flavor stability the oil requires hydrogenation beyond a shortening consistency, and hence would have to be blended with other more stable liquid oils to produce a high grade shortening.

## RESEARCH AND MARKETING ACT INVESTIGATIONS

### COTTON LINT

#### Fundamental Characteristics of Cotton Fiber as a Means of Developing New Uses (RM:s-102)

#### Method of Measuring the Cross-Sectional Swelling of Cotton Fibers

Knowledge of the moisture relations of fibers is of fundamental importance in all of the industrial uses of cotton where change in dimension is a factor in either processing or utilization. For the first time we have a reliable method for measuring the cross-sectional swelling in fibers. Estimates obtained in this way show relatively small varietal differences in cotton. Since several of the modified cottons produced here do exhibit large differences in swelling behavior, it is of importance to determine these differences quantitatively.

Knowledge of the salient points in the greater closing capacity of immature over mature fibers has been gained which will be useful in the development of self-sealing fabrics. Factors which appear to explain better closing capacity of the immature fibers are the smaller interfiber spaces due to the fact that there are more than twice as many fibers in a given weight of immature as in a like weight of mature fibers; the flatter and more irregular shapes of the thinner-walled fibers which makes for better packability; and the greater deformability of the immature fibers.

#### Oxidation of Cotton Cellulose

A better understanding of the mechanism of oxidation of cellulose will aid in preventing degradation of cotton fabrics. Research on this subject has clarified oxidation with acidic hydrogen peroxide, which is an oxidation closely related to that occurring in overbleaching where peroxide is employed. Another type of damage to cotton arises from the sensitivity of certain types of oxidized cotton to alkalies, even such weak alkali as soap solutions. The stabilizing action of chlorous acid and diazomethane against this type of sensitivity has been further investigated and has been shown applicable to two different types of oxidation.



In RMA contract research at the National Bureau of Standards no typical infrared spectra for carbonyl groups were found in oxidized cotton cellulose, indicating the possibility of their existence in all abnormal form. This concept provides a new approach to the direct measurement of degradation in oxidized cellulose.

#### Chemical Bonding of Cotton Fiber with Resins

The successful treatment of raw cotton with ethylenimine in vapor form without adversely affecting subsequent carding and spinning operations indicates the possibility of vapor phase polymerization of other substances in cotton fiber for the purpose of introducing firmly bound, uniformly distributed resins.

#### Reduction of Crystallinity of Cotton Cellulose

We have succeeded in reducing the high crystallinity of cotton without loss of fiber structure and without chemical degradation. The irrecoverable elasticity or stretch has been increased by the amine treatment. To increase elastic recovery it will be necessary either to introduce blocking groups or cross linkages by further chemical treatment. We have found that formaldehyde is a possible reagent.

#### Applications of Differential Dyeing

The differential dyeing technique has been successfully applied to cotton mill manufacturing and dyeing problems with savings in the purchasing of cotton and in the incidence of factory seconds. It is already in use also for maturity determinations in mills for the selection of cotton according to degree of maturity with respect to specific end uses.

The dyeing has been shown to be of potential value to cotton breeders and growers, particularly to those interested in the development of the cotton fiber on the plant.

Development of New and Improved Products from Cotton

Fiber Through Processing and Chemical Treatment (RM:s-104)

#### Cotton in Blends with Other Fibers

A survey report on blending has been made which provides a basis for an analysis of the following: (1) Whether there are any avenues of approach where cotton, in combination with other fibers, would improve its competitive position either by regaining lost markets, wholly or partly, or by finding new outlets; and (2) whether cotton would risk losing current and potential fertile markets by the proposal of types of blends which ordinarily would not have been envisioned. Conclusions reached in such an analysis will be used in determining types of investigations to be carried out later with



respect to blends of cotton fibers with other fibers in yarns, and blends of cotton yarns with yarns made of other fibers in folded yarns and fabrics.

#### Improving Insect Repellency of Cotton Bagging

The method of protecting cotton flour and feed bags from insect penetration by treatment with a mixture of pyrethrins and piperonyl butoxide, a synergist for pyrethrins, was made public in a Department of Agriculture press release July 25, 1949. A more detailed description of the process was given in a joint publication of this Laboratory and the Bureau of Entomology and Plant Quarantine.

Practical application to flour bags of this protective treatment is being tested by the Textile Bag Manufacturers Association. Flour was packed in the treated bags and stored for several months. The insect-repelling efficiency of these bags is being tested, and the flour analyzed for traces of contamination by the protectants. Incomplete results indicate an unexpected amount of migration of the protectants into the bag contents, resulting both in contamination of the flour and a lowered resistance of the cloth to penetration by insects. This behavior makes it necessary to seek formulations containing lower percentages of high-boiling organic liquids, as well as pyrethrin synergists and anti-oxidants of a waxy nature that will less readily leave the cloth.

Cloth has been treated at laboratory for trial as insect-proof liners and covers for bins and barrels used for storing grain. It has been reported that at least one large bag manufacturer is offering treated bags for use in storing seed grain.

#### Effect of Twist on Weaving Efficiency and Quality of Woven Fabrics

Weaving cost is 40 percent or more of the manufacturing cost of woven cotton fabrics. A major factor of this cost is loom operating (weaving) efficiency, which is significantly influenced by warp yarn twist. Yarn properties directly influenced by twist which effect weaving efficiency are (1) tensile strength, (2) elasticity, (3) resistance to abrasion, and (4) fatigue life (progressive failures). The results of twist studies to date have provided the basic guide for machine processes and adjustments for speeds, drafts, roll settings and twists for the manufacture of warp yarns with a high level of physical properties for weaving laboratory samples of a narrow width of a 40-inch, 4.00-yard, 80-square print cloth.

#### Contract Research Investigations

Improved cotton warp yarns, improved luster in cotton, control of neps during manufacture and improved resistance to soiling are the



subjects of research being conducted by contract with private and state research organizations. Of particular interest is the discovery that treating cotton garments with carboxymethyl cellulose in the wash water greatly increases resistance to soiling.

## OILSEEDS AND OILS

### Improved Methods of Cottonseed Oil Extraction, New and Improved Products from Cottonseed (RM:a-103)

#### Composition of Cottonseed as Influenced by Environment and Variety

The 3-year study of the composition of cottonseed as influenced by environment and variety will be completed when the samples from the 1949 season are analyzed and the data evaluated. The investigation is based on 8 selected varieties grown at 13 stations and on additional varieties grown at 4 of the stations. Included in the analyses are the oil content of the kernel, iodine number of the oil, gossypol, and trace metals.

#### Fractionation of Cottonseed Meats

The most important findings in the process development research on the fractionation of cottonseed meats are those on flake drying; cost analysis of the fractionation process; the application of a close circuit disintegration-screening operation to increase yield of fine meal essentially free of pigment glands, oil, and hulls; and the further reduction of the free gossypol content of the fine meal by conditioning operations incorporated along with desolventizing operation. Samples of pigment glands and different meal fractions, which were produced in the pilot-plant experiments, have been provided other research organizations for investigation of nutritional and toxicological properties.

#### Improved Screw-Processing of Cottonseed

An extensive investigation in cooperation with industry has been made of the variables involved in screw-press processing of cottonseed meal. It has been shown that the properties of the meal are influenced by the cooking and pressing conditions. When the conditions of cooking are maintained mild, the nitrogen solubility of the meal is determined by the energy input to the screw-press as related to the throughput. It was found that adequate oil yield can be obtained without undue energy input into the press and therefore it was found possible to produce cottonseed meals which had much higher nitrogen solubilities than those hitherto produced.



### Useful Products from Pigment Glands

The new compounds of gossypol with peanut protein, amino acids, dextrose, and starch have been made and given to a number of pharmacologists for determination of their effect on experimental animals. They have been shown to be lethal to goldfish in concentrations of 1 part to 100,000 whereas gossypol itself is not lethal at all and pigment glands which contain gossypol in its natural state are not quite as toxic as the new compounds. When these compounds are stored at elevated temperatures, pigment changes take place similar to those that occur in stored cottonseed. The new compounds provide a powerful tool for the investigation of the physiological action of gossypol as it occurs in the natural state, and of the changes which take place in gossypol and the gossypol pigments on storage of the seed.

### Investigations of the Nutritive Value of Cottonseed Meals

It was shown that the nutritive value of cottonseed meals containing low quantities of gossypol is related in some manner to the nitrogen solubility of the meal. This discovery provides an opportunity for continued systematic research on the relationship between the chemical properties of the meal and its nutritive value and provides a clue for the development of a chemical test which will provide an estimate of nutritive value of the meal.

An examination of the chemical properties of commercial cottonseed meals showed that there was considerable variation in the gossypol content of these meals and their nitrogen solubility. This demonstration has convinced the cottonseed industry and nutritional investigators alike of the inadequacy of the present methods of labeling cottonseed meal for feed purposes. It has convinced the cottonseed oil industry that research must be done on the effect of processing on the properties of the meal and has therefore enabled this Laboratory to obtain considerable cooperation from industry for this purpose. At the same time this demonstration has proved to nutrition investigators that they ought to re-examine all known information on the nutritive value of cottonseed meal. New tests were therefore planned, using cottonseed meals which had been analyzed and which had been produced under controlled processing conditions. In the first series of tests, approximately 5 tons of specially processed meal were provided to nutritionists.

Nutrition tests performed on these cottonseed meals demonstrated that screw-pressed cottonseed meal produced under mild conditions of cooking is as good as soybean meal as a source of protein for chicks and hogs. No evidence of toxicity was found even when these meals were fed in concentrations as high as 70 percent for chicks and 43 percent of the diet for hogs. This demonstration is in variance with the generally accepted viewpoint that cottonseed meal in large concentrations in the diet, is not suitable as a source of protein for chicks and hogs.



Quality Improvement of Peanut Products and New  
Uses for Peanut Oil (RM:a-124, Part I)

Research Aids Peanut Butter Industry

The separation of the oil from the meal phase in commercial peanut butters containing seemingly proper amounts of hydrogenated (hardened) peanut oil for the prevention of this undesirable characteristic was investigated and the mechanism by which such a stabilizer prevents oil separation was ascertained. It was determined that the maximum "stabilizing" effect is achieved only when the original crystalline condition of the stabilizer is not altered by any condition during or after its incorporation. Specifically, it was determined that the temperature of incorporation of hydrogenated peanut oil should not appreciably exceed the melting point of the hard fat.

This information has aided the peanut butter industry because it permits the selection of an adequate stabilizer on the basis of existing processing conditions and it indicates the processing conditions which should be maintained during and following the addition of hydrogenated peanut oils to achieve maximum freedom from oil separation.

Uses for Rice and Rice Byproducts and  
Methods of Processing (RM:a-21)

Although rice bran represents a potential source of approximately 50 million pounds annually of high-quality edible oil, the production of rice bran oil on a commercial scale in the United States has occurred only within the past few years. During the early efforts to process bran in a variety of solvent-extraction plants many unforeseen problems arose. These problems involved rapid deterioration of the bran during storage and difficulties as a result of the finely divided state of rice bran. Inability to refine the crude oil because of the presence of excessive quantities of phosphatides, waxes, pigments, and other as yet unidentified substances seriously threatened the economic development of this new industry. Many of these problems have been solved or the difficulties appreciably minimized.

It would seem from the experiments completed thus far, that micro-organisms play a minor role in the formation of free fatty acids in rice bran and all efforts in stabilizing this material must be concentrated on inhibiting the activity of the natural enzymes in the rice bran itself. Once this is done, there should be a relatively minor contribution to deterioration from any micro-organisms on the bran.

Storage experiments were conducted to determine a means of inhibiting the deleterious action of the enzyme, lipase, whereby rice bran



might be stored and accumulated to merit an economical extraction. Investigations were undertaken to obtain data on the naturally occurring active agent(s) responsible for deterioration of the bran. Based on these studies it was found that rice bran, which heretofore could not be stored for more than a few days, can now be stored for several months.

A successful means of degumming rice bran oil has made possible the production of a highly superior edible oil and has also led to the recovery of a highly useful byproduct, namely, rice bran wax.

The advantages to be gained by processing rice bran are production of a more stable bran of improved nutritional value and recovery of an oil adaptable to a wide variety of uses in the edible field, including cooking and deep-fat frying, manufacture of mayonnaise and salad dressings, and the manufacture of shortening and other plastic fats. The very marked resistance of the hydrogenated fat to oxidative rancidity is especially characteristic of rice bran oil.

#### Expanded Research on Fats and Oils (RM:s-556)

##### Research on Monoglycerides

Monoglycerides are used widely as emulsifiers for oil-water mixtures, which may be basic, acidic, or neutral in nature. Since it is known that the resistance of monoglycerides to decomposition is sensitive to changes in the acidity of their environment, the exact degree of resistance under various conditions is a matter of practical importance. The experimental work on monoglyceride emulsions has provided useful information on this aspect. For somewhat similar reasons, the results obtained with regard to the stability of monoglycerides at high temperatures are important. Relatively high temperatures are encountered when monoglycerides are used in the preparation of such unrelated products as baked goods and synthetic resins.

Sulfated monoglycerides, such as the short-chain fatty acids found in coconut oil, are sold in large quantities as household and industrial detergents; but similar products obtainable from cottonseed oil are unknown and have been of little importance heretofore. The work on sulfated monoglycerides and on continuous liquid-liquid countercurrent purification of technical-grade monoglycerides promises to solve some of the problems on the production and utilization of a variety of monoglycerides. The availability of monoglycerides of high purity undoubtedly will encourage the use of these compounds in new products and processes.

Hydrogenation of Fats and Oils

It is difficult to exaggerate the importance of the hydrogenation reaction in the processing and utilization of fats and oils. Between 1 and 2 billion pounds of fats are hydrogenated annually in the United States -- largely by empirical methods. The work in progress and reported here is not only of value in elucidating the mechanism by which hydrogenation proceeds; but it also makes possible the exercise of more precise control over the properties of the hydrogenation products. The results obtained to date on the hydrogenation of methyl oleate are especially valuable to the fat and oil industry in controlling the formation of "iso" acids during hydrogenation.



WESTERN REGIONAL RESEARCH LABORATORY  
M. J. Copley, Director

ALFALFA UTILIZATION INVESTIGATIONS

Growth-Regulating Factors

Satisfactory methods have been devised for the quantitative determination of the stereoisomers of beta-carotene in alfalfa meal. These isomers, designated all-trans, neo-B and neo-U, have relative vitamin A potencies of 100, 53, and 38, respectively. Application of the method to dehydrated alfalfa meal has shown that dehydration causes extensive isomerization of all-trans-beta-carotene to the other forms. Total carotene determinations which neglect this effect may give calculated vitamin A potencies which are too high by 15 percent. Irradiation of dehydrated meal reverses the isomerization to some extent.

Studies are under way in collaboration with State Experiment Stations on the probable existence of growth-inhibitors in alfalfa meal. Hot-water extraction has failed to reveal an inhibitor in either the extract or the extracted material.

Studies on the vitamin B<sub>12</sub> content of alfalfa as determined by paper chromatography have shown that more than 80 percent of the potency indicated by microbiological assay is due to factors other than B<sub>12</sub>. Chick growth tests have confirmed this conclusion.

Chemical Constituents of Alfalfa

Two or more naturally occurring antioxidants in alfalfa have proved to be good preservatives for carotene in mineral oil. Studies are under way on their isolation and characterization.

Water-soluble and water-insoluble fractions of the proteins of alfalfa have been assayed for amino acids and found to be well balanced, with little difference between the two fractions.

Studies now completed have shown that the optimum moisture content for retention of carotene in alfalfa meal in unsealed containers is 8 to 10 percent.

## RESEARCH AND MARKETING PROJECT

### Preservation of Carotene in Alfalfa Meal

Seventy different antioxidants have been tried as stabilizers for carotene in alfalfa during storage. The antioxidants are applied by spraying solutions of the compounds on alfalfa meal in a rotary mixer. They are tested by storing at 65°C. for 14 days. Five compounds (two substituted hydroquinones and 3 quinoline derivatives) have proved sufficiently effective as antioxidant stabilizers of beta-carotene to justify tests for possible toxicity.

## FRUIT UTILIZATION INVESTIGATIONS

### Freezing Preservation

Fundamental studies of the browning of peaches have revealed correlations between tendency to brown and tannin and enzyme contents. This information may be useful in estimations of dosage of antioxidant necessary, not only for peaches but for other fruits prone to darkening.

Efforts to improve acid-peeling methods for peaches have revealed certain properties of wetting agents in combination with acid solutions. The knowledge obtained will be useful in the application of wetting agents to processing operations. Wetting agents failed to correct the failure of acid solution to remove peel from certain green areas of fresh peaches.

Efforts to increase the firmness and the drained weight of thawed strawberries following preservation by freezing have included tests with added citrus pectins, of both high and low methoxyl contents. Sugar packs with various proportions of sucrose, dextrose, and invert sugar have been included. Results thus far have revealed no appreciable increase in firmness.

The development of information on a suitable wax mixture for application to berry picking boxes, with exact treating procedures, and more recently of information on design of treating equipment, has aided the berry industry. The wax not only reduces mold growths but extends the usable life of the boxes from one season (or less) to as much as three.

Studies on the effect of calcium as a firming agent for yellow Newtown apples stored 10 months showed that 500 ppm. of calcium in the sulfiting bath caused satisfactory firming of slices. A rapid and simple method for the determination of calcium content of dipping solutions has been developed.



Experiences of a commercial processor of cold-processed fruit spreads preserved frozen, which were developed by the Western Regional Research Laboratory, revealed difficulties with sucrose hydrate, which consists of mold-like, spherulitic agglomerations of sugar. Low temperature studies have shown that use of 30 percent of invert sugar in replacement of added sucrose, as well as packaging in hermetically sealed containers, are effective methods of preventing the formation of sucrose hydrate during storage and retailing of the products. Crystallographic studies on levulose have revealed a method of purification of this sugar. A rapid and accurate method of determining levulose in fruit has been developed.

Coliform bacteria, including Escherichia coli, have been found in about a third of samples of frozen concentrated orange juice in recent studies and this fact has significance from the point of view of sanitation. The work under way is of keen interest to the citrus processing industry, who cooperate through a committee on which the Western Regional Research Laboratory has representation. This Committee is considering the adoption of procedures used at this Laboratory as tentative methods for collaborative work. High-frequency radiation is being applied to cultures of E. coli to determine whether it has a specific (nonthermal) lethal effect.

A steam-injection stripper or preheater developed for use in recovery of fruit essences, has been found useful as a rapid sterilizer or pasteurizer of heat-sensitive products. In a test on apple juice heavily inoculated with a heat-resistant yeast, the "Thermilizer" sterilized the juice. The juice was heated to 244°F. and returned to 80°F. within a period of 0.6 to 0.7 of a second. This method has the minor disadvantage that it introduces some water into the juice.

#### Dehydration

A process for the preparation of a prune powder from commercially dried prunes (involving brief cooking, separation of pits in a finisher, drum drying, and grinding or flaking) has appeared to have commercial possibilities. An industrial group interested in prunes has obtained a supply of powder, prepared in this Laboratory, for market testing. In the course of the study a system of air jets was installed to aid in removal of the film from the drum; the technique proved satisfactory. Drum drying appeared to be superior to other methods tried, such as air drying, vacuum, and spray drying, particularly in costs. Estimated cost of production was 3.7 cents per pound of product.

Powders prepared with several varieties of figs also have interested commercial producers of that fruit. In addition, experiments on addition of fig powder to breads and breakfast foods have suggested that new outlets of this type may be commercially feasible.

Since caking of packaged fruit powders may be associated with extent of crystallization, studies are under way on kinetics of crystallization of amorphous sugars and also on a spectrometric method of measuring amounts of crystalline material in spray-dried powders and sugars. Relationships of storage temperature and moisture content to sticky points of fruit powders are also under investigation, beginning with methods of measuring moisture in the powders.

#### Dehydrofreezing

Success of frozen concentrated fruit juices, increasing costs of transport and storage, and the possibility of obtaining a more easily handled frozen product have all seemed to be reasons for increasing interest in dehydrofreezing. This method, if applied successfully in commerce, would reduce space requirements by at least 50 percent. Pilot-scale equipment will soon be available for a processing line for dehydrofrozen apples.

#### Canning and Bottling

Exploratory processing studies and cost estimates have been made on several products prepared from fresh prunes (French variety). The results of these studies have stimulated industrial consideration of the products (clarified and unclarified juices, puree, and juice concentrate) as new outlets for fresh prunes.

Studies on steps in processing the Washington variety of raspberry have pointed out factors in washing and grading that appear to be the cause of mushiness in the canned product.

Experiments on low-grade Delicious apples will be undertaken to supply facts on range of quality of processed juice obtainable, including such factors as acidity and its adjustment, blending with other varieties, and addition of ascorbic acid.

#### Pectin

In pilot-plant studies on the manufacture of low-methoxyl pectin from dried citrus peel, two new procedures have improved the process. Preliminary pickling of dried peel in dilute acid followed by drainage to recover solubilized pectin and by countercurrent extraction of the residue



to recover further pectin has proved superior to direct countercurrent extraction of unpickled peel. The second improved procedure consists of fine grinding of dried peel and concurrent extraction of pectin with dilute acid under controlled conditions of time and temperature. These procedures result in increased quality and/or quantity of pectin extracted.

Studies on industrial uses of pectic substances have included the following applications: coatings for seed corn, broken almonds, candied fruit mixes, dates, and fruit pieces for use in bakery products. All have been successful with the exception of seed-corn coatings, and further tests on that application will be conducted.

Several types of coating machine, involving spray coating, slot-orifice with falling film of pectinate, fluming, and flooded-curtain application, have undergone testing. The latter, in which the almond piece or other product passes through two heavy separate falling curtains of pectinate, seems to be most promising.

#### Grape By-products

A compound from grapes with high so-called "vitamin P" activity has been studied in an unsuccessful effort to determine its chemical structure. If toxicity studies prove favorable, further studies may be undertaken.

Samples of whole grape pomace have been supplied to the U.S. Bureau of Mines with the suggestion that they be tested for use in oil-drilling muds. Some tests have revealed that the pomaces had about 50 percent of the effectiveness of quebracho for this purpose.

#### Torula Yeast Studies

Pilot-plant studies have revealed pertinent information on the propagation of Torula yeast. The "off-fermentation" observed in earlier studies is actually a phenomenon related to foam formation, and may be avoided by adequate mixing in the propagator to disperse the heavy foam. Air supply and dispersion are critical in the growth of Torula; below about 1.2 cfm. of well-dispersed air per pound of yeast, activity is largely fermentative. At 1.2 or more cfm. of well-dispersed air per pound of yeast, the yeast grows rapidly and consumes sugar efficiently in the production of new yeast. With Torula, a diluted feed is unnecessary in starting a propagation. Propagations started on full-strength feed have proceeded at a rate near the known maximum for the organism.

### Vitamin B<sub>12</sub>

By means of screening studies, a strain of Bacillus megatherium has been identified as an efficient producer of vitamin B<sub>12</sub> activity. This organism has been grown on a 150-liter scale, with yields of 0.8 mg. of vitamin B<sub>12</sub> activity per liter of culture medium. Cultures of this organism have been distributed widely and the method is now undergoing commercial trials.

### RESEARCH AND MARKETING ACT PROJECTS

#### New Uses of Deciduous Fruits to Prevent Waste

A recent experimental development in pilot-plant equipment for the recovery of fractions of juices containing the aroma and flavor substances (essences) may extend applications of the process to materials that are too heat-sensitive for other equipment. This development consists of the combination of a steam-injection preheater with a short tubular evaporator. The injection of steam into the juice or puree provides very rapid heating action, with only slight addition of moisture, and prevents fouling of heat-exchange surfaces in the short evaporator tubes to which the steam-injection unit is coupled, because of the high velocity over the heating surface.

The usefulness of the essence-recovery process was demonstrated earlier at the Eastern Regional Research Laboratory. With pilot-plant equipment, including the modification mentioned above, the Western Regional Research Laboratory has conducted studies on recovery, concentration, and evaluation of volatile flavor-aroma fractions from juices and purees of various Western fruits. With the purees it has been possible to remove 40 to 50 percent of the water, along with most of the essence, and thus to concentrate the puree about two-fold in solids content. Concentrations of the distillates to various degrees, including 1200-fold, have been obtained in special equipment. These studies have revealed facts of importance in the development of industrial concentrates.

Studies on comparative flavor intensities of 7 varieties of strawberries grown in the Pacific Northwest have revealed significant variations. This method may prove useful in selection of varieties; work will continue over several seasons.

Comparisons of several processes for the preparation of frozen apple juice concentrate have produced information of value to prospective processors. One product was a 6-fold concentrated stripped juice made 4-fold with fresh juice. Another was a 4-fold concentrate of stripped juice to which



the fresh fruit equivalent of concentrated essence was added. A third was a 6-fold concentrate made 4-fold with fresh juice and concentrated essence. Juices were concentrated in an all-glass laboratory vacuum evaporator, in which juice temperature was varied by 15° increments from 85° to 145°F. Only the latter temperature appeared to affect flavor adversely. The only difference noted with the lower temperatures was the gradual lightening of color as the temperature was increased. A 4-fold concentrate of stripped juice was prepared in commercial vacuum-pan equipment at a juice temperature of 115°F. and concentrated essence previously recovered from the juice was added. All products containing essence were superior to those with only fresh juice added. The vacuum pan appears to be suitable for commercial use and has the advantage of low cost as compared with low-temperature evaporators.

Four-fold concentrates of Western Concord grape have been made to permit comparisons of processing methods and of methods of preservation (freezing and canning).

#### Spray Drying Studies on Citrus Fruits

In experiments on spray drying of concentrated orange juice, addition of methocel before drying to 3-fold concentrate resulted in a satisfactory powder. The yield was similar to that of single-strength juice and feed rate on a solids basis was several times greater. Six-fold concentrates with added methocel also could be spray-dried satisfactorily, but the yield was lower than that of 3-fold.

Other drying aids besides methocel that have proved satisfactory are carboxymethocel and acetylated pectin. Relationships of sticky points to temperature and relative humidity were determined. Development of off-flavors and off-odors in the powders was found to be closely correlated with loss of carotenoids. In a search for a carrier for fat-soluble antioxidants that might be introduced, a new drying aid (glyceryl monostearate) was discovered.

#### Continuous Press for Recovery of Fruit Juices

A commercial company employed under a research contract has constructed a quasi-continuous, 6-cylinder, hydraulic press for use on fruits and fruit wastes. Single-cylinder tests have shown that the press will separate raw, milled pear waste into a juice fraction containing about 0.3 percent insoluble solids and a press cake suitable for drying. Minor changes are being made in the press in preparation for delivery of the unit to the Western Regional Research Laboratory.

### Conversion of Wastes into Feedstuffs

Studies in laboratories and in a plant at San Jose, Calif., during the 1949 season in collaboration with the Cannery League of California, have established the practical feasibility of a process for the separation of pear waste into fractions and also for further processing the fractions into molasses and pomace products. Further studies on various steps will be made in 1950, again with collaboration of the League. Products obtained in 1949 are being evaluated as feeds by the University of California. Cost studies based on data available thus far suggest that a business venture based on utilization of waste may be successful if allowance is made for present waste-disposal costs. Analyses for insecticide residues have been made on pear waste products that are being used in feeding trials.

In response to a request by officers of the California Prune Marketing Program, methods for the application of dyes to surplus and substandard prunes have been developed. The method consists of application of oil- and water-soluble dyes.

Evidence of the existence of an unidentified growth factor in tomato juice has accumulated in recent studies. The factor apparently is responsible for growth of a *Leuconostoc*-like bacterium and is widely distributed. Effort to identify it with known factors has been unsuccessful.

Under a research contract, the Colorado Agricultural Experiment Station has made studies of methods of ensiling sugar beet tops in order to preserve them for dehydration, and methods of dewatering the freshly harvested leaves in order to reduce the dehydration load. The objective of work under the contract is to determine whether it is feasible to produce a high-quality dehydrated feed from beet tops, which are now commonly left in the field when the beets are harvested.

### Fundamental Studies of Fruit Tannins

Work has been initiated to isolate, purify, and establish the chemical structure of polyhydroxyphenol substances (tannins) that serve as substrates for the enzymatic browning in peaches. Removal of the tannins from the fruit, and their partial purification and concentration, has been accomplished by a series of extractions using various organic solvents. Paper chromatography of an ethyl acetate extract showed that chlorogenic acid may be one of the components. Further work is in progress to separate and identify the components of the tannin concentrate.



## POULTRY UTILIZATION INVESTIGATIONS

### Freezing Preservation of Turkeys

Studies on relationship of the amount and composition of fat in the diet of turkeys to the fatty acid composition of the carcass fat, the frozen storage stability of the carcass, and the flavor of the meat have established the following points:

Important changes in carcass fat composition result from differences in diet fat of an amount and character which could normally be expected between practical feed formulas. Such changes in carcass fat composition have a marked effect on accelerated storage life of the fat, and hence probably play a decisive role in determination of the frozen storage life of the carcass.

Fat that turkeys on a fat-free diet deposit, which necessarily must be synthesized from carbohydrate or protein, is quite different from that deposited by turkeys on a practical diet. The synthesized fat is more saturated and has a composition more nearly like that of beef fat, or fat from other range-fed animals. It appears that the fat composition generally attributed to species of poultry is almost completely an expression of their diet and not of their specific metabolism.

Addition to a turkey ration of highly unsaturated oils, whether of fish or vegetable origin, results in objectionable fishiness in cooked meat. This quality is present in the freshly slaughtered bird, and apparently does not increase in intensity during frozen storage.

This fundamental information is of practical value to the poultry industry, because it emphasizes the relative importance of diet fat in the determination of quality and storage characteristics. Diet effects for turkeys have been removed from the field of qualitative conjecture to one of quantitative knowledge. A possible means of improving frozen storage quality through dietary modification has been demonstrated.

A study of warm versus cold evisceration of turkey hens in relation to their effect on quality and frozen storage stability has provided valuable information for poultry processors. From the standpoint of possible visceral taint in the eviscerated bird, there appears to be no danger involved in holding the uneviscerated bird for one day at 35°F., and the acceptability after frozen storage does not appear to be markedly affected. For

optimum stability of the carcass fat in storage, however, warm evisceration should be recommended because of the resultant elimination of the holding period above freezing.

Studies on keeping quality of frozen turkeys have demonstrated that if turkeys are well packaged and maintained at a constant temperature, the differences in acceptability, after one year of storage, between storage temperatures of  $-10^{\circ}$  and  $+10^{\circ}\text{F}$ . are small. This suggests that processors should look to packaging and other factors as a cause of storage failures.

Methods of preparation and storage of frozen turkey steaks have been evaluated and the necessary information to obtain a satisfactory stored product has been published. Technical information on the steaks is of direct value in promoting the utilization of turkeys in this form, and thus increasing consumption and providing additional marketing outlets for surplus turkeys.

### Egg Proteins

Studies were begun on the mechanism by which the thick egg white thins during the storage of shell eggs. The problem was attacked from three approaches: (1) Detailed studies were initiated on the preparation and properties of ovomucin, the egg white protein reputedly responsible for the firmness of thick white. Ovomucin preparations obtained appeared quite different from those reported in the literature. (2) The effects of chemical agents were studied. An old observation that sulfhydryl compounds would thin thick egg white was confirmed and extended to include other reducing agents. Minute amounts of thioglycol were found to cause rapid breakdown of other egg constituents. It was also demonstrated that reducing gases (hydrogen sulfide) or vapors (thioglycol) would penetrate through the shells of intact eggs to give effects simulating those found in badly deteriorated, storage eggs. (3) The origin of the cause of thinning was sought. It was considered possible that small amounts of reducing substances might diffuse into the white from the yolk during storage. It was conclusively demonstrated that an important cause of thinning is inherent in the egg white itself.

Studies were continued on three of the biologically active proteins, lysozyme, conalbumin and avidin. Conalbumin was isolated by a more satisfactory method and a number of derivatives prepared. Avidin was also prepared by a more satisfactory method. Work was initiated on the effects of specific hydrolysis of the phospholipids of yolk and yolk constituents.



### Frozen Creamed Chicken

A study of precooked frozen creamed chicken, inoculated with *Staphylococcus*, showed that delay in freezing after preparation is more harmful than delay in consumption following defrosting. Two hours' holding prior to freezing appeared to be maximal. Quickly frozen product can be defrosted over a period of 11 to 12 hours without excessive development of bacteria.

A study was made to determine the existence and extent of a lag phase in growth of bacteria following freezing and a period of holding in the frozen state. Cultures of *Micrococcus pyogenes* were used for this purpose. The results indicate that the spoilage "safety margin" for thawed frozen foods is greater to some extent because of the freezing. It appeared to be greater above 0°F. than below and also seemed to have doubtful practical significance.

Studies are also under way on the species of micro-organism that survive freezing and storage of turkey steaks, in order to extend basic knowledge of contaminants.

### Calcined Feathers

Application of the process of calcination to wet feathers (60 lbs. steam pressure for 30 minutes or 40 lbs. for 60 minutes, followed by drying and grinding) has produced an economically attractive meal worth \$60 or more per ton for use in mixed fertilizer. Estimated cost of processing is about \$35 per ton.

## RESEARCH AND MARKETING ACT PROJECTS

### Precooked Poultry and Other Dishes

The effect of the class and breed of chicken on the flavor of frozen creamed chicken has been studied with leghorn fowl and roosters and colored fowl, roosters, and roasters. The only consistent difference found was the comparative lack of flavor in the product prepared from the roasters. The older and less expensive chickens are, therefore, not only permissible in precooked frozen foods but will yield a more flavorful product, and their tenderness can be adequately controlled by the use of a suitable cooking method.

Experiments comparing creamed frozen turkey and chicken prepared under the same conditions showed a greater rancidity in samples containing turkey and turkey fat than in those containing chicken and chicken fat. Therefore, any

surplus of large-sized turkeys cooked by the usual method could not be utilized by substituting them for chicken in precooked frozen products without an adverse effect on the quality of the product. However, preliminary results show that the use of antioxidants during the cooking process holds considerable promise in solving the problem of turkey fat rancidity in precooked frozen foods.

The relative storage stability of precooked frozen creamed turkey, chicken, and peas was studied under varying conditions of packaging and temperature. The results show that with an excellent container (sealed can) storage temperatures in the range of  $-30^{\circ}$  to  $+10^{\circ}\text{F}$ . had no effect on off-flavor development. However, with a typical commercial container (MSAT cellophane liner, carton, and laminated waxed paper overwrap) the higher temperature accelerated off-flavor development. Thus, the use of an adequate packaging material has been shown to be important.

Since turkeys, unlike chickens, are not readily available throughout the year, it is at times difficult to obtain a freshly killed bird to serve as a standard of comparison for storage studies. For this reason a long-term storage study was initiated to obtain information on the stability of turkey held at  $-30^{\circ}\text{F}$ . After 8 months' storage no difference was found between the stored birds held at  $-30^{\circ}\text{F}$ . and the fresh controls.

Experiments on precooked frozen eggs have been designed to study and if possible to prevent the texture change in the cooked frozen white that has made eggs an unacceptable ingredient in precooked frozen foods. Freezing-rate studies have shown that a texture most nearly like that of the unfrozen cooked egg was obtained in samples that had supercooled <sup>before</sup> freezing. The determination of the mechanism of the change that occurs when cooked egg white is frozen will be the basis for further attempts to develop a satisfactory cooked egg for use in precooked frozen foods.

The relative storage stability of frozen blanched vegetables and frozen cooked vegetables has been investigated with peas, carrots, celery, lima beans, corn, snap beans, green peppers, water chestnuts, and mushrooms. Peas have shown greater changes in storage than the other vegetables, and in the three lots studied the cooked peas have deteriorated at a much faster rate than the blanched peas. However, when the cooked peas were mixed with white sauce before being stored, their rate of deterioration was slight and was essentially the same as that of the blanched samples.



## Preservation of Dried Egg

A rapid, practical, and inexpensive processing method was developed for the removal of glucose from egg material. This method consists simply in adding less than one percent yeast to the liquid egg material in vats and allowing the yeast to ferment the sugar within 3 hours. The glucose-free dried whole egg was found to have a shelf-life over six times that of the normal dried egg. Considerable interest in this product has been expressed by many sources, including the Food and Container Institute for the Armed Forces and various commercial producers and users of dried eggs. Studies on the chemistry of dried egg deterioration conclusively demonstrated that glucose reactions are the principal deteriorative reactions occurring during storage.

## VEGETABLE UTILIZATION INVESTIGATIONS

### Dehydration

The project on application of a starch coating to protect dehydrated carrots is being continued. Various laboratory and commercial samples of corn, wheat, and potato starches were equally successful in stabilization of color of dried carrot. Wheat starches with widely varying fluidity showed no differences. Ascorbic acid, ascorbyl palmitate, and hydroquinone solutions used as dips have not proved as valuable as a plain water dip for enhancement of color.

Studies on dehydrated potato have included (a) determinations of brown discoloration in samples heated at various moisture contents and temperatures, (b) storage studies on commercial potato powder, (c) investigations of suitability of dehydrated and dehydrofrozen potato for use as French-fried, shoestring, and chip potatoes, and (d) effects of metallic container contamination on color of diced potatoes.

Studies on drying rates of potato pieces in the low-moisture range (as in drying bins) have shown direct relationship to drying rate history at high moisture levels; that is, the density of the nearly dry piece is a factor which depends upon the previous drying history.

### Freezing Preservation

The effect of variety of peas on temperature tolerance in frozen storage has been under investigation. Rates of change in color, flavor, and texture are being established by these studies.

The flavor-bearing constituents of peas are being studied by analytical methods. Data on pod storage of peas are being obtained; for example, deterioration after 6 days at 35°F. was slight--less than after 2 days at 77°F. For reference standards it has been found necessary to store peas at -30°F. rather than -10°F.

A new calcium-carbide weight-loss method for determination of moisture in sweet corn has been developed. It is rapid and comparatively simple and will probably prove reliable for the determination of maturity in sweet corn.

In studies of inactivation of enzymes of vegetables by scalding, complete inactivation of peroxidase (and possibly slightly less than complete inactivation) has been found adequate for sweet corn, lima beans, and Brussels sprouts. Contrary to the general rule, it was easier to inactivate peroxidase than catalase in lima beans. Holding vined lima beans in ice at 33°F. for 24 hours prior to freezing resulted in no serious loss of quality, whereas a delay of 4 hours or more at 75°F. caused impairment. Immersion in ice water beyond 5 hours caused deterioration.

Studies on regeneration of peroxidase in scalded cauliflower and peas suggest that delay at high temperature may result in regeneration following scalding. Investigations on Brussels sprouts indicated that residual peroxidase actually decreases during a delay between scalding and freezing.

Frozen cream-style corn has proved to be an attractive product. Only whole-kernel sweet corn is frozen commercially.

#### Canning and Other Processing

Froth-flotation cleaning developed in earlier research at the Pullman, Washington, laboratory has been successfully applied to sweet corn in midwestern states for removal of borers. The investigations will continue during the 1950 season.

The process of canning food products with mild heat and small amounts of the antibiotic subtilin has been applied successfully to uninoculated packs of peas, asparagus, green beans, wax beans, tomato juice, mushrooms, sweet corn, cauliflower, broccoli, Brussels sprouts, rice, whole milk, cream, sole, and turkey in white sauce. Screening tests have been made on 14 antibiotics and of these subtilin seems to be the most promising. Studies are under way on mode of action of



subtilin against spores; early findings indicate that subtilin attacks the spores during initial stages of germination. In this study a rapid method of counting spores of Clostridium botulinum was discovered--consisting of addition of small amounts of sodium bicarbonate to the medium to stimulate germination.

Instrumental methods for the accurate recording of color of vegetables during processing have proved successful for frozen peas, dehydrated carrots, and lima beans. Microwave spectra are under investigation as a possible method for the identification of volatile constituents of vegetables.

#### Hop Constituents

Additional studies on lupulon and humulon, antibiotic constituents of hop cones, have included preparation of a number of derivatives in an effort to find compounds with antibiotic activity. One, hexahydrolupulon, has tuberculostatic activity approximately equivalent to that of lupulon. Kilogram quantities of lupulon and humulon have been prepared and samples have been distributed widely to research agencies. Clinical trials of lupulon and humulon on human tuberculosis have yielded inconclusive results. Lupulon is undergoing trial for treatment of leprosy in Louisiana. A spectrophotometric method has been developed for the estimation of lupulon in the blood stream of patients.

#### Freezing and Cooling Equipment

Further studies on immersion freezing have included studies on cans of food partially immersed in brine. Partial-immersion freezing may make possible the use of automatic equipment that is used in the canning industry for the cooling of cans. Rates of freezing for orange juice and solid products were practically the same for partial as for complete immersion.

Comparative studies on freezing rates of canned products in a refrigerated air blast showed that No. 2 cans of peas (16 oz.) froze in less than 75 percent of the time required to freeze 12 ounces of peas in paper cartons. The increased freezing rate would make possible some increase in capacity of existing air-blast freezers.

Operating tests with a new post-blanching cooler have shown that about 3 minutes is required to cool potato dice from about 200°F. to 80°F., with an air velocity of 1,450 feet per minute.

## RESEARCH & MARKETING ACT PROJECTS

### Uses for Rice and Byproducts, Processing and Handling

Steam treatment has proved to be a satisfactory method for the stabilization of brown rice against fatty acid development. Repeated tests have shown improvement in milling yield provided drying is adequate. Color is not changed. Greater adhesion of the bran coat makes longer milling necessary. Cool storage (32°F.) and low moisture content also control the development of fatty acids. Efforts are being made to obtain natural antioxidants from de-fatted rice bran.

A food product known as Rice Curls, recently developed, has stimulated considerable interest. The product can be made from broken grains and is essentially a deep-fat-fried snack food, formed by extrusion of a cooked paste through a small die.

Frozen cooked white rice has been demonstrated to be an attractive product from the point of view of quality and quick preparation. A quick-cooking puffed rice is undergoing development.

### Dried Beans and Peas

Substitutions of bean flour for wheat flour in bread have shown that taste is not affected up to 10 percent, but loaf volume is reduced noticeably with 20 percent substitution. Studies of varieties of beans involve rate of loss of palatability at various temperatures and relative humidities. A new variety, Pioneer, developed by the Washington State Experiment Station was found unsuitable for canning. Experiments on snack foods made from beans and peas are under way. Products suitable for quick preparation are also being tried--for example a dried preparation for use in soup.

Studies are continuing on the nature of the trypsin inhibitor in beans and on the relationship between inhibitor content and nutritional value. Basic studies on pea starches are also under way, particularly on the characteristics of starches of smooth and wrinkled varieties of peas and on amyloses and amylopectins.

Under this project a small-scale method for the isolation of starch and protein from wrinkled and smooth peas has been developed. The yields are fairly good, especially with smooth peas. Expansion of the scale of the method appears straightforward and should offer little difficulty. A new rapid method for analysis of



starch and amylose in peas has been developed and applied to about 100 samples. It has been found that separation of the genetic factors controlling wrinkledness of the skin and amylose content is difficult.

#### Edible Mushroom Mycelium Produced by Submerged Fermentation

Commercial interest in the production of mushroom mycelium by submerged fermentation continues. Objectives of current research are enhancement of flavor of the product by treatment after fermentation (to increase flavor and shorten time in the fermenter), isolation of additional strains of fermentable mushroom in an effort to find some that yield better flavor, and further studies of nutritive and environmental conditions for optimal production of flavor.

#### Conversion of Potatoes to More Stable Forms and Products

Investigations related to the pre-peeling process for white potatoes (for sale ready for use) have included development of an objective method for measurement of changes in texture, tests with acidified sulfite solutions as dipping agents for retention of color, and experimental storage of cooked riced potato. All of these studies have yielded promising results.

Dehydrated diced potato, prepared by freezing cooked potato and then dehydrating it, offers encouragement as a quick-cooking product. It is porous, readily absorbs water, and makes attractive mashed potato, potato cakes, soups, etc. A new type of French-fried potato consists of extruded mashed potato and potato flour, which is fried in deep fat; it can be preserved by freezing.

#### Measurement of Maturity in Peas

An extensive survey of literature on the maturity of peas and on methods of measuring maturity has been completed. A new method for the determination of tenderness and maturity, involving vacuum treatment to remove air beneath skins and flotation in sugar solution (rather than brine) has aroused interest among inspection officials and others. The method is less tedious and more reliable than the one specified in U.S. Grade Standards.

#### Sugar-beet Processing Liquors

Research on composition of sugar-beet processing liquors has included diffusion juice, thick juice and molasses from four factories in the Western region at

two different times during the harvesting season. While little difference was observed in the seasonal samples, there was a higher concentration of non-sucrose impurities in the liquors from California than elsewhere. Only twenty to thirty percent of the impurities are removed during processing providing an incentive to develop new purification procedures. This composition study will be expanded to include all the major production areas of the country.

Methods for identification and fractionation of organic acids by means of differential absorption have been developed.

Studies on betaine indicate that derivatives are likely to be an uneconomical outlet for betaine and that uses requiring the unchanged material should be found. A new method developed for betaine analysis is of considerable interest to sugar beet analysts and poultry feed producers.

#### WHEAT UTILIZATION INVESTIGATIONS

##### Gluten Phosphate

Additional improvements in the production process for gluten phosphate (a gel-forming substance derived from wheat gluten) have resulted in a tentative production-cost estimate which is substantially lower than the cost of earlier processes. The new cost is \$1.65 per pound. This modification consists of use of 70 percent acetone rather than water in the washing step; thus the bulk of the wet product is decreased and drying is simplified. The cost estimate is based on the assumption that 98 percent of the acetone is recoverable.

##### Baking Characteristics

In continued studies on the influence of various flour fractions on baking characteristics, both glutens and buffer-soluble constituents were found to vary among flours in contribution to baking behavior. With many flours, variations in soluble constituents appear to overshadow differences in glutens. Effects of fractions of solubles have also been studied; the soluble pentosans, for example, had little, if any, effect on characteristics of baked bread, but did affect the character of the dough. The crude albumin fraction gave much the same volume-increasing effect as total solubles; thus it seems that soluble proteins are of importance to loaf volume.



## Milling Characteristics

In studies of milling qualities of various wheats, evidence obtained indicates that a relatively high crude fat-crude fiber ratio in whole wheat indicates poor milling quality (as exemplified by the Rex variety).

## WOOL UTILIZATION INVESTIGATIONS

### RESEARCH & MARKETING ACT PROJECTS

#### Fundamental Studies to Develop New and Improved Uses

One phase of the fundamental studies on wool involves electrical properties including the dielectric constant in the microwave region, another the crystalline-to-amorphous ratio by X-ray diffraction, and still others involve application of physical-chemical methods in studies of wool and mohair as a basis for modifications that may increase usefulness. Progress has been made in these various phases.

Modification of wool by beta-propiolactone in a vapor-phase treatment has a stabilizing influence on reduction of the disulfide bonds between main protein chains. Stress-relaxation studies of the kinetics of disulfide-bond reduction indicate that both beta-propiolactone and alkenyl succinic anhydrides have this stabilizing effect.

Ultracentrifugal and diffusion studies of wools in solutions of urea have shown that most of the wool can be broken down into mixtures of small fragments, the molecular weights of which are about 12,000 and that wools of varying fineness and origin vary only a little in this respect. In the process of carbonization wool is treated with strong acid to destroy vegetable impurities. A study of acid degradation of wool has shown that an induction period exists in which damage can be held to a minimum. The hysteresis behavior of wool has been found to be correlated with adsorption of agents known to interact with hydrogen bonds.

Under contract to the Princeton Textile Research Institute (and a sub-contract covering processing to the Forstmann Woolen Co.) four master wools have been classified, sampled, and scoured. One has been carried to the top stage of processing and a measurable aging effect has been demonstrated. A major aim of the studies is to determine whether aging during processing affects spinnability as the basis for more effective utilization of our domestic coarser grades of wool.

Various analytical studies have been applied to the samples, such as amino-acid analyses, alkali solubility, phenol adsorption, coefficient of friction (by the Western Regional Research Laboratory), stress relaxation and reflectance (by the Princeton Textile Research Institute) and fineness and length distribution and scouring (by the Forstmann Company).

#### Methods of Scouring Wool

A promising laboratory method has been developed that cleans grease wool through the utilization of the detergent properties of natural suint soaps present in the raw wool. The detergent properties of these natural soaps are greatly enhanced by addition of small amounts of alcohols and neutral electrolytes. The wool grease can then be made to separate and the dirt to settle by further addition of alcohol. The alcohol is finally recovered by distillation and can be returned to the system. Further study of this process will be necessary in order to evaluate such variables as temperature, rates of movement of solvent and grease wool, distillation rates, and other factors which would be important in operation on a larger scale.



EASTERN REGIONAL RESEARCH LABORATORY  
P. A. Wells, Director

APPLE AND OTHER FRUIT UTILIZATION INVESTIGATIONS

JUICE PRODUCTS

New Portable Essence Unit

Construction of an improved pilot-plant apparatus for investigations on the recovery of essence ("volatile flavor concentrates") from fruit juices, or from distillates obtained in the evaporation thereof, was completed. This new unit has a capacity of 10 gallons per hour of juice at 10 percent vaporization or 5 gallons per hour at 20 percent vaporization. It is of portable type, so that it can be transported in a station wagon to any desired site of operations. The principal improvements in function over equipment previously used are (1) the provision of a preheater ahead of the vaporizer tube, which prevents the surging that has sometimes been found troublesome, (2) the use of chilled column bottom effluent to scrub the vent gas for more complete recovery of essence than by the scrubbing with chilled essence hitherto employed, (3) the minimal size of all parts carrying hot juice, which (at the 10 GPH rate) has reduced from about 20 seconds to 2-1/2 seconds the time that the juice is exposed to possible heat damage, (4) the provision of a holding coil enabling the increasing of this time if and as desired. Thus there can be studied the effect, on flavor and aroma, of heating time, which for some juices is harmful and for others may actually result in an increased yield and/or quality of essence.

Recovery of Concord Grape Essence During Vacuum Evaporation

Preliminary to a detailed study of the recovery of volatile flavors given off during vacuum evaporation of fruit juices, some tests were made on pasteurized Concord grape juice. After depectinizing the juice, it was evaporated to a concentrate, using a vacuum of approximately 27-1/2 inches. The vapors were condensed in a surface condenser using tap water at 46° F., the resulting condensate having a temperature of 57° F. No attempt was made to recover the volatiles in the vent gases leaving the condenser. The condensate was then put through the new portable essence recovery unit mentioned above, vaporizing approximately 50 percent. The recovered essence was incorporated in the proper proportions into the concentrated juice and sufficient water added to restore the original volume. When this reconstituted product was compared with the original pasteurized juice and with diluted depectinized concentrate the following observations were made by a research taste panel.

The fully reconstituted juice (containing the essence) was far superior in taste and aroma to the concentrate reconstituted with water alone. As compared with the starting juice, the sample containing the recovered essence was somewhat deficient in both taste and aroma. The results, therefore, show that the vent gases from the surface condenser should be treated, by scrubbing or otherwise, to recover the aroma they contain.

## Market Development

On August 17, Bill H.R. 5270, to exempt the volatile fruit concentrate or essences from the Internal Revenue Tax provisions, became a law. On September 27 the regulations for the production of these essences were published in the Federal Register and became effective immediately. Since this latter date commercial interest in both the production and applications of the various fruit essences has been greatly accelerated. The general opinion of those actually engaged in the manufacture of essences and those contemplating its manufacture is that the regulations appear to be rather too stringent. However, activity in the commercial production and utilization of fruit essences has proceeded at a greatly accelerated rate in recent months.

## COMPARISON OF APPLE VARIETIES FOR PIE BAKING

Further flavor studies were made in the calcium chloride firming of the softer summer and early fall varieties of apples by comparing them with the standard firm winter varieties. The following 15 varieties were used: Summer varieties - Yellow Transparent, Williams, Rambo, and Gravenstein; early fall varieties - Dutchess, Wealthy, McIntosh, Grimes Golden, Jonathan, and Red Delicious; winter varieties - Golden Delicious, Stayman Winesap, York Imperial, Stark, and Baldwin. Canned Baldwin slices and frozen Baldwin slices were again used as comparative standards for the canned and frozen series, respectively. All samples were baked into pies and rated for flavor by a taste panel of 12 persons, using a scale of 10-1 for scoring, 10 being best.

By calculating the standard error of difference between two averages of control and calcium chloride treated slices, it was found that the use of calcium chloride for firming did not significantly affect the flavor of the apple slices.

No variety, canned or frozen, was found to be significantly better than the Baldwin standard. Of the frozen slices, Yellow Transparent, Williams, Rambo, Gravenstein, McIntosh, Jonathan, Golden Delicious, and Stayman Winesap were not significantly different from the standard; Dutchess, Wealthy, Grimes Golden, York Imperial, and Stark were the poorer varieties. By groups the frozen slices averaged as follows: summer varieties - 6.4 untreated, 6.6 treated; early fall varieties - 6.2, untreated, 5.7 treated; winter varieties - 6.0. Only canned Dutchess slices were poorer than the canned Baldwin standard. The average scores of canned varieties by groups were: summer, 5.4 untreated, 5.5 treated; early fall, 5.3 untreated, 5.5 treated; winter, 5.7.

## VEGETABLE UTILIZATION INVESTIGATIONS

### VEGETABLE WASTES

## Processing Studies

A process for preparing high-grade vegetable leaf meals in through circulation apron dryers was published in AIC-76 and Technical Bulletin No. 958. This process has not found wide industrial adoption principally because



apron dryers are inherently costly and are not widely used in farm operation. On the other hand, direct heat rotary alfalfa type dryers are cheaper, have high evaporating capacity, are simple to operate, and are widely used for processing farm crops such as alfalfa. For this reason we purchased a Heil 2-pass unit with an evaporative capacity on alfalfa of 2,500 pounds of water per hour. A preliminary report was made last year on the use of this dryer for the fractional drying of broccoli for the production of a high quality broccoli leaf meal. During the past year similar drying studies on a variety of vegetable waste materials were completed.

The results clearly indicate that vegetable wastes can be successfully dehydrated in this manner. The wastes studied were pea vines, broccoli, lima bean vines, spinach field waste and beet tops. Whenever the availability of sufficient waste permitted, three inlet air temps were investigated, namely, 850-900° F., 1050° F., and 1200° F.

For comparative purposes a portion of each waste was dried at 240° F. in a through circulation dryer. On the basis of carotene retention and carotene recovered in leaf meal per ton of waste, high temperature rotary drying compared very favorably and at times was better than through-circulation drying at moderate temperatures. In the case of pea vines the recovery of carotene in the leaf meal by the rotary dryer method is nearly twice that of the through-circulation dryer method. For lima bean vines and broccoli, carotene retention was about equal for both types of drying at the lower temperature of about 900° F. However, the destruction was only slightly higher at the intermediate temperature of about 1050° F. and would be preferable over the lower temperature due to increased capacity. Drying at 1200° F. was accompanied by an appreciable destruction of carotene.

Spinach and beet tops show a higher destruction of carotene in the rotary dryer at all temperatures investigated. In the case of spinach, carotene retention was approximately 15 percent lower in the rotary dryer for all three temperatures. However, the meal was of very high quality, having three times as much carotene and twice as much protein as a typical alfalfa meal. Furthermore, the economic advantages to be found in the rotary dryer method over the through-circulation dryer method would indicate the choice of the former despite the somewhat lower carotene recovery.

### Feeding Trials

The feeding trial, "A Comparison of Broccoli Meal and Soybean Oil Meal as Protein Supplements in Broiler Rations," was completed at the Delaware Agricultural Experiment Station. In this trial solvent extracted broccoli leaf meal was used as the protein supplement in a diet for comparison with soybean oil meal to determine rate of growth and feed efficiency. A standard University of Delaware broiler ration and a 50-50 broccoli-soybean mixture were also included in the trial. Vitamin supplements (including the new B<sub>12</sub> or animal protein factor) were supplied in adequate amounts.

The data showed that extracted broccoli leaf meal was not as good as soybean oil meal or a standard broiler ration in promoting growth in chickens. This is probably due to the slightly higher percentage of basic amino acids in the soybean meal and broiler ration. Although the chickens on broccoli meal were smaller, they were more perfectly feathered and of

generally better appearance than the all soybean birds or the standard ration birds. The best all around group of birds was the 50-50 soybean-broccoli fed group. The weight of this group was about equal to the soybean and standard ration groups and the feathering and general appearance were better. It is very unlikely that there will ever be sufficient extracted broccoli leaf meal to allow its use as a replacement for soybean meal in high energy broiler rations but combined with soybean meal it should be a valuable supplement since it appears to supply some factor missing in the soybean meal. Further investigations will be made to determine what this factor might be.

#### Growing Broccoli for Feed

In the summer of 1948 a cooperative project was initiated at the Virginia Agricultural Experiment Station on the growing of broccoli for use as a feed supplement. During the summer of 1948 small experimental plots were prepared using different broccoli varieties and different spacings to determine the best conditions for growing this material for preparation of a high grade leaf meal. Some of this work was described last year. At that time it appeared feasible to cut this broccoli top growth two or three times during a season rather than harvesting the entire plant at the first cutting.

Larger scale plots were planted in the 1949 season and the results have recently been sent to this laboratory by our Virginia co-workers. The top growth on these larger plots was cut with a mowing machine instead of by hand and a yield of better than twelve tons of green top was obtained per acre. The twelve tons of green material would yield slightly under one ton of dry leaf meal. This result is consistent with our previous experimental work at King Farms Company in Morrisville, Pennsylvania.

In contrast to the small hand harvested plots of the 1948 season the yield of second top growth was much smaller than the yield of the first cutting. No third cutting was made. Some of this work will have to be repeated under more favorable growing conditions than were found during the dry summer of 1949, since broccoli cannot withstand long periods of drought.

#### SAPOGENINS FOR THE PREPARATION OF ANTI-ARTHRITIC COMPOUNDS

The discovery of the anti-arthritis action of cortisone, pregnenolone, artisonone, and other steroids has led to an intensive search for suitable precursors for these compounds. Steroidal sapogenins are potentially one of the best precursors for anti-arthritis. Consequently the Bureau of Agricultural and Industrial Chemistry and the Bureau of Plant Industry, Soils, and Agricultural Engineering have engaged in a cooperative search for the best plant sources of these compounds. Since January 1, twenty-two different lots of plants collected in North Carolina, Virginia, Arizona, Panama Canal Zone, and Puerto Rico have been received.

#### TOBACCO UTILIZATION INVESTIGATIONS

##### Tobacco Essence and Flavor

An investigation was made to develop a method for the recovery of tobacco essence from low-grade tobaccos and tobacco stems. Such an essence, it is



believed, would be used to enhance lower grade tobaccos or impart a tobacco aroma to paper, wood or similar items used in the sale of tobacco products.

Aqueous and organic solvent extraction methods were studied. Compared with water leaching or steam stripping, extraction with an organic solvent is more efficient. Tobacco essence is obtained as a residue after distilling off the solvent from a Soxhlet extraction of ground tobacco stems. Alcoholic solutions of the residues were impregnated into filter paper and evaluated organoleptically for quality. Ethanol extraction produced the most representative essence concentrate with tert-butanol, methanol, acetone, chloroform, benzene and iso-propanol next in order of preference.

The effect of Soxhlet extraction time on essence recovery was studied. A twelve hour extraction is required for complete recovery. A similar study on batch-wise extraction at varying temperatures revealed that a two- to three-hour extraction at 78° C. is required to give a comparable extract.

When comparable methods of extraction were employed on both leaves and stems, the leaves gave a better quality and stronger extract than the stems, although the difference was not great. The nicotine content of various essence extracts was determined. An average of 89 percent of the nicotine available in the tobacco is found in the extract. Tobacco treated with a denicotinized extract was milder than that treated with a nicotine containing extract but was inferior in aroma.

Ten varieties of cigar tobacco stems and three varieties of cigarette type stems were extracted and evaluated for desirable aroma. The varieties were rated in the following decreasing order: Connecticut Broad Leaf, Connecticut Shade, Red Burley, Imported Cuban, Imported Cuban Wrapper, Pennsylvania Filler, Flue Cured, Havana Seed Leaf, Light Burley, Wisconsin, Sumatra, Ohio Broad Leaf and One Sucker.

Several tobacco manufacturers in the New York City area have been canvassed concerning their interest in tobacco essence. One important cigar manufacturer expressed a real interest in securing test samples of cigar tobacco essence for flavoring flattened cigar stems which they are using in a low priced cigar. They also manufacture low priced smoking tobaccos of high stem contents and are anxious to test Burley and Flue-Cured essences in these products. Several others, manufacturing smoking tobacco and cigarettes of low nicotine content, are desirous of testing Burley and Flue-Cured essences if they are nicotine free. In processing tobacco for their low-nicotine products they remove some of the aromatics from their tobaccos and they would like to be able to restore a natural tobacco flavor to their product. A nicotine-free essence appeared to them as just the material they are seeking.

Everyone expressed an interest in testing the essences as "top flavors" to give a fine tobacco aroma when the package is opened. Also as an additive to the material making up the package or box in which they pack their products. This latter, however, appears to be more difficult as it would probably have to be incorporated into the paper or wood. Staining was suggested as a possible objection, and there is some question regarding retention in the paper for any great length of time.

## PHOTOCHEMICAL DECOMPOSITION OF NICOTINE

Last year we reported some results on the photochemical oxidation of nicotine. One of the reaction products, a di-(nicotine dioxide) appeared to be identical with a substance isolated from fermented cigar tobacco by another investigator. During the past year further work was done to elucidate the character of the photochemical oxidation products obtained from nicotine. Chromatographic analysis indicates that there are at least six substances produced during the oxidation, and of these three occur only in small amounts. Similar analysis of the material from fermented cigar leaf indicates the presence of five or six components. Although as yet positive identification of the compounds has not been made, the evidence points to a similarity in the composition of these products with those obtained by photochemical oxidation.

As reported last year, if cultures of Newcastle disease and influenza virus were photo-oxidized to completion, total inactivation of the viruses was observed. However, at this stage the antigenic properties were also sacrificed. In view of the high impurities of these preparations further work on phages will be limited to those which can be obtained in high purity.

In order to obtain a better understanding of the photochemical reaction on proteins, the oxidation of crystalline lysozyme and beta-lactoglobulin in the presence of traces of methylene blue was investigated. Preliminary results indicated that the decrease of enzymatic activity could be correlated to some extent with the decrease of tryptophane and tyrosine content of the enzyme. Correlation was also extended to the remaining two amino acids, histidine and methionine, which are photochemically strongly reactive. When lysozyme was photo-oxidized to the extent of 2 mol. of oxygen per mol. of enzyme, the total histidine content was gone, while the enzyme activity decreased 70 percent. Methionine concentration of the enzyme was not affected even after 16 mol. of oxygen uptake. It would appear that histidine in the lysozyme molecule represents the major active group, tryptophane to a lesser degree, while tyrosine would assume a questionable minor importance. Methionine by itself does not act as an active group in the enzyme molecule. Similar experiments carried out on crystalline beta-lactoglobulin yielded essentially similar results. Solubility studies carried out on this protein during photo-oxidation showed that the decrease in solubility was parallel to the decrease of the tryptophane content of the protein.

On the basis of these experiments it appears that sensitized irradiation of these two proteins preferentially oxidizes first histidine, followed by the oxidation of tryptophane (with some overlapping) while the oxidation of tyrosine proceeds at a much slower rate. In spite of the high reactivity of methionine alone, it is highly resistant to photo-oxidation if incorporated in the protein molecule.



## MILK PRODUCTS UTILIZATION INVESTIGATIONS

### PROTEIN INVESTIGATIONS

#### Bristle Fiber from Milk Proteins

Factors involved in the cost of production of casein bristles were explored. An important factor in the cost of production is the amount of fiber produced per hour by an extrusion unit. The Eastern Regional Research Laboratory experimental unit extrudes four pounds an hour, which is the rate of extrusion obtained commercially. It has been found that the amount of casein carried by the extruder screw is the principal factor limiting production and not the number of holes or volume of openings in the spinnerette.

With the cooperation of a local commercial firm, it was demonstrated to parties interested in the production of casein bristle that high rates of fiber extrusion can be attained. Using a twin-screw extruder at a screw rate of 30 R.P.M., 26 pounds of casein were extruded, and at 70 R.P.M., 45 pounds of casein were extruded per hour. Experiments on the laboratory extruder showed that the rate of extrusion could be increased 50 percent by changing the design of the screw.

Large quantities of casein bristles have been made for testing as a padding material, particularly in furniture. The coiled fiber is being tested by one furniture manufacturer, as well as by several local upholsterers. The commercial manufacturer of coiled casein fiber states that he has obtained trade acceptance for its use in upholstering.

#### Separation of Caseins

Previous reports have described the separation of the principal constituents of commercial casein, namely alpha- and beta-caseins. Electrophoresis experiments demonstrated a slow moving component, gamma-casein, present in quantities of 2 - 4 percent in unfractionated casein. A method for separating electrophoretically pure gamma-casein was devised.

Studies on the physical properties of pure gamma-casein show that in some respects it resembles beta-casein. The marked difference of gamma-casein in phosphorus content, isoelectric point and the action of rennet is particularly noteworthy. It is possible that gamma-casein is similar to beta casein in composition other than the phosphorus content.

### CARBOHYDRATE INVESTIGATIONS

#### Recovery and Purification of Lactic Acid

Ammonium lactate. The report for last year described the esterification of ammonium lactate with various alcohols and the production of n-butyl lactate in high yield by recycling the by-products. A company interested in the utilization of surplus citrus molasses adapted our results on the esterification of ammonium lactate with butanol to pilot plant operation.

Obtaining good results, they are now building a plant capable of producing 4,000 gallons of butyl lactate per week. The manufacture of butyl lactate from ammonium lactate by this method should provide this lactic ester at lower cost, stimulate the utilization of lactic acid, and offer a larger outlet for inexpensive sugar-containing materials.

Extractive Esterification. A new approach to the problem of recovery of lactic acid from aqueous solution involving the simultaneous esterification and extraction of aqueous lactic acid solution by various alcohols is being investigated. In this process, a mixture of aqueous lactic acid and a water immiscible alcohol is refluxed in the presence of an esterification catalyst. Some esterification occurs and the ester, as well as the acid, is distributed between the two phases.

Extractive esterification is promising because the acid, being highly water soluble, is difficult to purify efficiently by solvent extraction. By carrying out the extraction under conditions permitting esterification, the alcohols become more efficient extractants, and hence solvent extraction of lactic acid becomes a more attractive commercial process, both as a means of purifying lactic acid, and also as a means of preparing the higher lactic esters from dilute aqueous solutions.

Plasticizer Studies. The esterification of butyl lactate with adipic acid produces a mixture of esters highly efficient as plasticizers for vinyl chloride copolymers. The same mixture of esters was also produced readily by esterification of adipic acid, lactic acid, and butanol or by esterification of a mixture of adipic acid, methyl lactate, and butanol. Thus lactic acid or methyl lactate when used with butanol was equivalent to using butyl lactate in the esterification reaction.

This mixture was examined by the Dow Chemical Company as a plasticizer for Saran, a vinylidene chloride copolymer. They reported that preliminary tests with this plasticizer were encouraging. Large scale evaluation tests are now under way.

#### Compounding and Properties of Lactoprene EV

A method for preparing Lactoprene EV blown sponge has been developed. Preliminary tests have been conducted by the firm of Burgess-Berliner Associates, and they pronounce the product to be distinctly superior in heat resistance and hot compression set resistance to materials heretofore used for the intended purpose. The product is designed for use in industrial fabric pressing machines. The Eastern Regional Research Laboratory recently supplied the Burgess-Berliner Associates with some special test pieces of the sponge designed to fit the smaller commercial presses, which they propose to test under production conditions. They have stated that if the projected tests confirm earlier laboratory results, they plan to recommend this material for the application described. They claim the application to be one requiring substantial quantities of sponge.



## ANIMAL FATS AND OILS UTILIZATION INVESTIGATIONS

### Basic Composition of Animal Fats

Work has been continued on the investigation of glyceride structures and compositions of a number of common animal fats with the special view of developing a practical process for separating glyceride fractions that have potential application as special purpose fats. Following previous work (of an analytical character) on glyceride fractionation as a basic guide, attempts have been made to make the fractionation more practical by reducing the solvent/fat ratio to a minimum without too great a loss of fractionation. Solvent ratios from 40:1 (original method) to as low as 5:1 have been tried on several animal fats without gross changes in the character of fractionation. Analysis of fractions are being made to define the extent of changes in the separation. Determinations of their chemical and physical characteristics are also being made. It is anticipated that some of these fractionated fats (of inedible grade) will be evaluated for use in hot dip tin plating.

### Stability and Storage Investigations

Work on the deactivation of metallic contaminants in lard has been continued. From a study of the relative pro-oxidant activity of the four most common metallic contaminants, it was found that 0.2 parts per million (p.p.m.) of copper ion is equivalent to 2, 4, and 20 p.p.m. of iron, nickel, and tin ions, respectively, in lowering the stability of lard from 8 hours (AOM) to about 1/2 hour. It was found that the addition of 0.01 percent of ascorbic, citric, tartaric, or phosphoric acid partially or in some cases almost entirely counteracted the pro-oxidant effect of these metallic ions in lard in the concentration mentioned. When some of the most powerful antioxidants were added to lards containing these amounts of metallic ions the effectiveness of the antioxidant was decreased markedly, and in some cases completely nullified. Ferric ions were generally more destructive to the antioxidant property than were the other metallic ions in the concentrations studied.

The results on metal deactivation studies not only clearly emphasize the need for packers to take extreme precaution in the manufacture and processing of fats and oils in order to minimize metal contamination, but also suggest a possible means of counteracting the pro-oxidant effect of traces of metals that may unavoidably be present.

The results also show the necessity of modifying present methods of determining rancidity in a fat when studying the effect of varying amounts of metals in a fat. Apparently a comparatively high concentration of metals in a fat not only has a pro-oxidant effect, but also causes a decomposition of the peroxides that are formed in the oxidation process, thus affecting the analytical procedure.

### Synthetic Detergents from Animal Fats

Since present commercial sulfated alcohols and sulfated monoglycerides are derived from coconut oil, a program was started to make sulfated alcohols and sulfated monoglycerides from animal fats or their fatty acids and to evaluate the products.

## Unsaturated Amides

The preparation of a new class of unsaturated amides has been undertaken. A number of the compounds have been prepared, and with the exception of the dimethallyl amides, once crystallized amides were of high purity and were obtained in 89 to 92 percent yields. These amides are potentially useful as monomers in polymerization or copolymerization reactions and in the preparation of surface-active reagents and other derivatives.

Beef fat has been converted to unsubstituted amides and glycerol by reaction with ammonia under pressure for six hours at 165°- 175° C. Glycerol and mixed amides were obtained in substantially quantitative yield (97 to 98 percent). Crystallization of the mixture of amides from a hexane fraction does not give a satisfactory separation of amides into saturated and unsaturated portions.

The ease with which beef fat can be converted to glycerol and unsubstituted amides at relatively low pressures and temperatures and in a short time may be of value where the mixed amides can be used without separation, such as in the preparation of waxes, water-repellents and surface-active agents. If the amides can be separated by a simple crystallization procedure, the process would be of still greater value.

## POTATO UTILIZATION INVESTIGATIONS

### Styrenated Allyl Starch

A reproducible method has been developed for copolymerizing styrene and allyl starch and producing a modified lacquer of outstanding resistance. Although it is intended to make a more complete study of the various proportions of allyl starch and styrene that can be used, present data favor equal amounts of each, on a double-bond basis.

Coatings of excellent resistance have been obtained by the styrenation of allyl starch in the presence of small amounts of soybean oil, dehydrated castor oil, and various amounts of allylsucrose. Styrenated allyl starch solutions in toluene-butanol have a higher tolerance than comparable allyl starch solutions for hydrocarbon diluents such as cyclohexane and turpentine. This fact might make the copolymer lacquer more satisfactory for the shelf goods trade.

Baked films of allyl starch-styrene copolymer have considerably better resistance than allyl starch films. Air-dried films of the copolymer, as presently formulated, have only slightly better properties than air-dried allyl starch.

## CHEMICAL AND PHYSICAL PROPERTIES OF POTATO STARCH

### Sloughing of Potatoes

Previously it was shown that excessive sloughing of potatoes on cooking was associated with high densities of the original tubers. Continuing these experiments, a sample of Green Mountain potatoes was separated into



sloughers and non-sloughers by differential flotation in brine of specific gravity 1.090. Tubers which floated did not slough when boiled, whereas tubers which sank did slough. However, after several months of cold storage, none of the tubers sloughed excessively, although they had not changed in density. If, however, these tubers were permitted to stand at room temperature for two weeks, the high density tubers regained their capacity to slough. This observation pointed out a limitation in the method of separating sloughers and non-sloughers by flotation in brine, while at the same time it helped to define more exactly the conditions under which the method might be valid.

The general observation has been made that the extent of sloughing varies with the starch content of the tissues, sloughing being greatest in tissues of the highest starch content. Within every sample of tubers studied, this relationship has been confirmed. Furthermore, sloughing can be decreased by decreasing the starch content of tissues, or increased by increasing the starch content of the same tissues. This is done by altering the storage conditions of the tissues. Recently, new data have been obtained which account in part for the unequal extent of sloughing from tubers of the same density and same storage history. It was found that such tubers may differ in starch content, and that the starch content, as previously, determines the extent of sloughing. For example, in tissues of the same density and storage history which had average "soluble solids" contents of 7.3, 7.8, 8.3, and 8.8 percent, the respective sloughing losses were 20, 12, 8 and 4 percent. In these cases the "soluble solids" content (which is inversely related to the starch content) indicated the relative starch concentration.

On the other hand, experiments in which tubers were stored at 50° and 75° F. indicate that there are factors other than starch content which affect significantly the degree of sloughing. The degree of sloughing may vary while the starch content, percentagewise, remains constant. This variation seems to be associated with physiological changes in the tubers. There may be changes in the properties of the starch, or changes in the cell sap which limit the swelling of starch granules when heated. In stored tubers, especially in those stored at a relatively high temperature, the proportion of inorganic salts to starch gradually increases, owing both to the loss in moisture and to the decrease in quantity of starch. This change would tend to limit the swelling of starch when heated and to diminish the sloughing.

Furthermore, during storage the originally distended cells lose moisture and shrink. This effectively increases the thickness of the cell walls and intercellular cement. At the same time the starch content of the cells decreases quantitatively while the number of cells remains constant. Thus the ratio of cells to starch increases, and the swelling of starch during heating would be less likely to distend and separate the cells and cause sloughing. Changes that would be expected to occur in the pectin during potato storage would promote rather than diminish sloughing.

## CONVERSION OF POTATOES TO STABLE FORM

### Drying of Ground Potatoes for Flour and Feed in a Direct Heat Single Pass Rotary Dryer

Tests were completed on the drying of ground raw potatoes in a direct heat, single pass rotary dryer for the manufacture of flour. The process proved successful in that the color of the product remained satisfactory after repeated recycling, mixing and drying operations. The possibility of using a higher moisture content in the feed to the dryer was also investigated and it was found that balling of the material occurred in the dryer at higher moistures. A moisture content of from 42 to 45 percent was found to be optimum.

In all tests  $\text{SO}_2$  was added at the rate of .075 percent based on the weight of the raw potatoes. This quantity is the same as is recommended in the steam tube dryer method. However, in the direct heat rotary dryer method this quantity proved to be in excess of that required to provide a maximum of 500 PPM in the final product. (Limit specified by the Commodity Credit Corporation). In these cases the  $\text{SO}_2$  in the product averaged 970 PPM which was probably due to the appreciable quantity of  $\text{SO}_2$  present in the products of combustion that come in direct contact with the potatoes being dried. Therefore, no specific quantity of  $\text{SO}_2$  can be recommended to be added to the raw material, since it would depend on the sulfur content of the fuel used in each specific instance. Work will be continued to adapt this method to multi-pass alfalfa type dryers during the coming season. The foregoing process can be cheapened to produce feed by elimination of the sorting and sulfiting steps.

## TANNING MATERIALS, HIDES, SKINS, AND LEATHER INVESTIGATIONS

### DEVELOPMENT OF NEW AND POTENTIAL TANNING MATERIALS

#### Sumac

Studies on the tannin contents of wild sumacs, which have covered a number of years, have been published in USDA Technical Bulletin No. 986, issued in December 1949. This bulletin reports on the tannin contents and abundance of 8 species of sumac that grow wild in the eastern and southern parts of the United States.

Data in the bulletin obtained from an intensive survey of the quantity of sumac growing wild in an area of approximately 12,000 square miles in the southernmost tier of counties of Virginia indicate that about 43,000 long tons of dry sumac leaf would be available there annually. This survey is probably the only estimate of collectible sumac ever made by careful field measurements.

The data indicate that Rhus copallina, R. glabra, and R. typhina are most promising for commercial development and that R. trilobata might prove of value under certain circumstances. The remaining four species, R. aromatica, R. microphylla, and R. virens, have objectionable features that

R. lanceolata



would probably prevent their successful competition with the other four species.

### Hemlock Bark

The Michigan College of Mining and Technology is interested in the utilization of hemlock bark from hemlocks of the Upper Peninsula of Michigan. If found to be economically feasible, consideration will be given to the erection of a commercial tanning extract plant. A survey of available bark supplies, cost of a plant of minimum economic size, production costs, and amount of hemlock extract that can be absorbed by the tanning industry are being studied by a chemical engineer of the Forest Products Research Division of the above College.

To aid in this program for the development of more adequate supplies of domestic tannins, the Laboratory has furnished pertinent information on hemlock bark, extract and tanning extract manufacture. Two samples of hemlock bark have been examined which showed tannin contents of approximately 13 percent, and extract purities of over 70 percent.

Samples of bark from dead trees show an unusually low nontannin and high purity. This may be the result of the slow leaching out of nontannin after the tree died. These samples both show tannin and purity which indicate that they should be very satisfactory for use in the production of a high grade tanning extract.

The survey has indicated that the minimum economical extract plant would have a capacity of 12,000 cords of bark per year and would produce about 1,000 tons of 100 percent hemlock tannin. In 1946 the Upper Peninsula of Michigan could have produced 125,000 cords of bark if all logs cut had been peeled. However, only 8,000 cords of bark were sold. It thus appears that a production of 12,000 cords of bark per year, an amount sufficient to operate one extract plant, would be possible.

### Saw Palmetto

Two samples of saw palmetto were analyzed for tannin and showed the following results, on a moisture-free basis, for the samples collected above and below ground:

	<u>Tannin</u>	<u>Purity</u>	<u>Total Sugar</u>	<u>Starch</u>
Above ground	8.4	39.1	5.4	13.9
Below ground	8.3	39.7	4.2	17.9

Water extractions were difficult due to the presence of starch. Extraction with 50 percent acetone in water gave lower tannin but higher purity of extractive. Fermentation of the water extractive removed most of the sugars and raised the purity of the extracted liquors, but purities were still below those of good tanning materials. Diastasing the materials before extracting lessened the mechanical difficulties of extraction but apparently resulted in a material loss of tannin. The purities of the extracted

liquors were lowered and the sugar contents were raised to 19.4 percent for the sample above ground and 22.5 percent for the sample below ground. Because of the relatively low tannin content (about equal to that of chestnut wood), the very low purity and the difficulty of extraction, saw palmetto does not appear promising as a source of tannin.

#### ALUM RETANNAGE OF VEGETABLE-TANNED LEATHER

The alum retannage of vegetable-tanned leather for the production of more serviceable insole leather has proceeded to the point where commercial tanners are showing considerable interest in the process. Production of satisfactory alum retannage of vegetable-tanned insole leather has been conducted in cooperation with a large shoe company. In a previous test 200 hides had been retanned for insoles, using a solution of aluminum acetate added to the oil wheel in the regular tannery procedure. This gave satisfactory results except for a somewhat cracky grain. A further test was made with 200 hides. In this case the aluminum acetate was added dry to the mill. In an attempt to cut down crackiness, the amount of  $Al_2O_3$  used was cut about 50 percent, and the leathers were thoroughly scoured before retannage to remove uncombined material in the grain. The solid material was thoroughly taken up by the leather. However, the takeup was not uniform as analysis showed that some leathers contained more  $Al_2O_3$  than others. The procedure reduced the crackiness of the grain somewhat but not entirely. Further tests are to be made for this purpose.

Laboratory studies have been made to produce a cheaper form of aluminum acetate solution by the reaction of calcium acetate and aluminum sulfate with decantation of the aluminum acetate solution from the precipitated calcium sulfate. Results so far have been promising for cases in which a solution may be used.

#### INVESTIGATIONS CARRIED OUT UNDER THE RESEARCH AND MARKETING ACT

Work Project No. RM:a-87 - Study of the chemical, physical and biochemical properties of apiary products for the purpose of extending their utilization, of developing from them new and improved products, and of finding new uses for them.

Dried Honey-Skim Milk. Our goal in this investigation is to develop completely dried combinations of milk (especially skim milk) and honey, containing at least 50 percent of honey solids. Successful products have been made in which the ratio of honey solids to skim milk solids reached 60:40; and in one case, using whole milk, to 42:58. Eighty-five pounds of a dry 50:50 mixture of skim milk and honey solids was prepared at the Bureau of Dairy Industry, Washington, and shipped to this laboratory for further processing and evaluation of possible uses. Considerable difficulty has been encountered with caking of the product in storage. It has been found that the moisture content below which the material does not cake varies with the particle size. This problem is being further studied.



Flavor Removal. Two processes for treatment of low-grade strong-flavored honeys have been developed. The first is a mild treatment which removes only the stronger flavor components, leaving the honey with its characteristic floral-type flavor, resulting in only minor color reduction. Bentonite treatment of the diluted honey affords a possible means of "standardizing" dark honeys for baking use, resulting in a product with considerable flavor, controlled pH and considerably reduced colloid content.

The second process involves dilution to 50-55 Brix, liming to pH 4.1-4.4, treatment with activated carbon, filtration and reconcentration to honey density. The product in this case is a bland, light-colored, high-density sirup with no honey flavor. A process run using 3,000 pounds of 1948 buckwheat honey was observed at an eastern honey cooperative.

Crystallized Honey-Fruit Spread. In order to avoid marketing de-flavored honey in competition with low-cost invert sugar, work was done to develop products commanding a higher return. It has been found possible to prepare what is believed to be a new fruit product, using de-flavored honey as a base. This product is a fruit spread which does not depend on the action of pectin for its consistency. By evaporating mixtures of de-flavored honey and fruit (juice or puree) to a high density and subjecting this to a controlled crystallization, a smooth, fondant-like, non-sticky fruit spread is obtained. This is analogous to Dyce-processed honey. Products have been successfully made from peach, strawberry, red raspberry, apricot, and cherry purees, from grape, loganberry, pineapple juices, from grape juice concentrate and from frozen orange juice concentrate. The products from grape, pineapple, and loganberry are especially fine.

Work Project No. RM:a-113 - Disposal of dairy wastes

Work during the past year showed that the primary process in aerobic oxidation of dairy wastes is an extremely rapid assimilation of the organic matter into microbial cell solids. It was found that one-half of the organic nutrients are almost instantly assimilated, the other half being simultaneously oxidized to carbon dioxide and water to gain energy for the growth process of the organisms. If the high oxygen demand of this coupled reaction system is met no acid is produced; essentially the only reaction product is the bacterial cell.

These results explain the previously puzzling failure of other investigators to reduce by aeration the oxygen demand of milk waste more than about 50 percent as a maximum. They also explain the production of acid from milk wastes in conventional sewage systems, for the latter process is an anaerobic alternative metabolic path for the organisms. The use of additional aeration when such wastes are being handled in conventional systems is obviously indicated. Development of a process designed to favor this assimilation and the subsequent removal of the organisms appears promising.

Work Project No. RM:a-125 - New and improved uses of deciduous fruits especially to prevent waste

Biochemistry of Sour Cherries. A cooperative study with the Michigan Agricultural Experiment Station on handling and processing sour cherries

proceeded along two lines of activity: (1) a study of the effect of various spray treatments on the quality of sour cherries, and (2) the effect of various pre-processing practices on the yield and quality of processed fruit. Since ferric dimethyldithiocarbamate (Fermate) and wax emulsion sprays are believed to produce low soluble solids fruit, it is imperative to establish the effect of these spray materials on soluble solids and the effect of the latter on drained weight. The results thus far indicate that the principal effect of the sprays is to delay maturity. When the various plots were picked on the same date, the Fermate and wax sprayed cherries were lower in soluble solids and lighter in color than copper sprayed cherries. When the various spray plots were compared at the same stage of maturity, the soluble solids contents were nearly identical for copper and Fermate sprayed cherries. Wax caused a significant decrease in soluble solids.

In a study of the behavior of bruised and unbruised cherries, sound fresh fruits were compared with those bruised by dropping from known heights, using a standardized procedure. Sound, unbruised cherries gain about 5 percent in weight but lose about 3 percent of their total soluble solids when soaked 24 hours in water at 50° F. The rate of increase is proportional to the temperature of the water. On prolonged soaking, for 2 or 3 days, the cherries increase steadily in volume to a maximum of about 10 percent and then split. Bruised cherries lose excessive amounts of soluble solids on soaking. They show little if any gain in weight and do not become turgid enough to cause them to crack. Bruised cherries are about 40 percent less firm than unbruised ones. Both bruised and unbruised cherries are firmed slightly by soaking in dilute calcium chloride solutions. Cooling, either in water or in air, causes cherries to increase in firmness.

Work Project No. RM:a-129 - Investigations of basic properties and functions of tannins and development of new tanning materials

Production of Canaigre. Field studies were conducted in cooperation with the Bureau of Plant Industry, Soils, and Agricultural Engineering. A study was conducted of drying of shredded canaigre under 12 different conditions to select a satisfactory procedure for use with the crop to be harvested in 1950. Analytical and laboratory leaching tests showed that shredded roots can be satisfactorily air-dried, when conditions are right, either out of doors or indoors on concrete, when spread at the rate of one pound per square foot of surface.

Over 30 tons of fresh roots were harvested in 1949 from experimental plots at Yuma, Arizona; 15 tons of fresh roots were shipped to the Eastern Regional Research Laboratory for drying and leaching studies and the balance was shredded and air-dried at Yuma, yielding approximately 4 tons of dried roots.

A considerable quantity of quebracho extract - the most important vegetable tanning material - is being used in regulating the viscosity of oil well drilling muds and is thus not available to the tanning industry for making leather. A sample of powdered canaigre root submitted to the Humble



Oil and Refining Company for laboratory testing was found to be effective in reducing the filtration rate of salt-water muds, and this company expressed an interest in conducting an actual oil well drilling test. To further check the suitability of this material, four tons of shredded roots at Yuma, Arizona have been hammer milled to powdered form and are now being tested by the Humble Oil and Refining Company as a possible replacement for quebracho tanning extract in the regulation of oil well drilling muds.

In preparation for experimental work to be done during 1950 approximately 12 acres of canaigre have been grown and are due for harvest in the summer of 1950. A yield of about 100 tons of roots of the "red" or high-tannin root type is anticipated.

For supplying a larger quantity of high quality roots for use in processing studies and the production, on a pilot-scale, of tanning extract sufficient for semi-commercial tanning tests, and to secure data on areas suitable for commercial growing of canaigre, the Bureau of Plant Industry, Soils, and Agricultural Engineering has established new canaigre plantings at Plainview, Texas; Portales, New Mexico; Elfrida, Chandler Heights and Higley, Arizona, totaling approximately 14 acres. These should be available for harvest in 1951.

Chemical engineering investigations have developed a more economical method of drying fresh canaigre. This consists of comminuting the fresh roots in a rotary knife cutter or a sharp knife hammer mill with 1/4" or 1/2" screens and drying in a direct heat rotary kiln dryer. With roots of the yellow variety, air at 500°-600° F. inlet temperature can be used without diminishing the tannin recovery on subsequent leaching after re-grinding. Up to 800° F., the recovery diminishes somewhat, and at 900° the loss is appreciable.

Processing Studies. Our continuous countercurrent extractor has been considerably modified and instrumented to make it more adapted to the leaching of canaigre. Eighty-eight continuous countercurrent leaching runs, each of at least 8 hours duration, have been made during the year to investigate the many variables encountered in this type of extraction. The solvents used included distilled water, 10 percent and 18 percent aqueous isopropyl alcohol and 25 percent aqueous acetone. From the results obtained, conditions suitable for large-scale extraction can be suggested.

Preliminary cost estimates have shown that the continuous countercurrent extraction of canaigre is the most feasible for commercial operation of any leaching process thus far studied for the recovery of tannin from this material.

Work Project No. RM:a-132 - Processing *Nicotiana rustica* as a source of nicotine

A process for the production of 40 percent nicotine sulfate from *Nicotiana rustica* has been developed and proven in a small integrated pilot plant. It is based partly on one of the commercial methods for obtaining nicotine from tobacco. The rustica juice is expressed from the plant by

a cane mill or a continuous rotary press, limed and stored in steel tanks. Throughout the year it is withdrawn from storage as required, clarified by heating with soda ash and filtering, extracted with kerosene in a continuous liquid-liquid extraction column and the kerosene extract treated with sulfuric acid to form the 40 percent nicotine sulfate product. This process has been shown to be operable both on freshly pressed juice and on year-old juice stored at room temperature. In the pilot-plant operations 85 percent of the nicotine from the fresh rustica plant was recovered in the product. While a somewhat higher recovery would be possible by refinements of this process, it would involve higher processing costs which probably could not be justified by the increased yield.

Work Project No. RM:a-152 - Improvement of quality and flavor of maple sirup and other maple products

High-flavored Maple Sirup. Based upon previous observations that color in maple sirup could be developed at will, dependent upon the time of heating at some given temperature above 190° F., a process has been devised for making a maple sirup having a concentrated or enhanced flavor and color. The process consists in heating maple sirup at atmospheric pressure at elevated temperatures for a definite period of time. The steps of the process are: (a) boiling the maple sirup until sufficient water has been removed to cause the boiling point to be raised to some temperature between 240° F. and 250° F; (b) holding the thickened sirup at this elevated temperature (three hours for fancy sirup and a lesser time for the lower grades) by preventing further loss of water through the use of a tight lid fitted with a reflux condenser; (c) adding water, when cooking is completed, equivalent in amount to that lost by evaporation, and allowing the sirup to cool. This sirup so treated develops a color and a concentration of flavor which is at least four-fold in respect to the starting sirup. One part of this concentrate can therefore be diluted with three parts of cane sugar sirup to yield a maple sirup blend that is practically indistinguishable from the original sirup. The process is simple and does not require any additional equipment to that found in a maple sirup processing plant, other than a kettle lid provided with a long upright pipe to serve as a reflux condenser. This process is applicable to all grades of maple sirup and does not cause appreciable development of caramel and other off-flavors in the lower sirup grades. Blends resulting from the use of this new highly flavored sirup will be rich in maple flavor and in color, and any off-flavors present in the original sirup will be diluted to an unobjectionable level.

Permanent Color Standards. Cooperative work with the Production and Marketing Administration on the development of permanent glass color standards for maple sirup was completed, and the new grading sets are commercially available for the 1950 season. About 210 sets of the new graders are in use by Federal and State inspectors and by private firms and individuals. A complete color grading set consists of three colored glass standards mounted in a metal box, three square bottles containing glycerin water "blanks," three square bottles containing standardized cloudy suspensions (for grading cloudy sirups), and empty square bottles for maple sirup samples.



Work Project No. RM:a-166 - A. Conversion of potatoes to more stable form and products  
B. New methods of using potatoes for feeds and non-food uses

Conversion of Surplus Potatoes to Stable Form. Last year we discussed a method for producing feed and flour from raw potatoes by grinding them and drying them in a steam tube dryer. Although the resulting flour was of a color satisfactory for the purpose intended, i.e., European export, it was inferior in color to that produced conventionally from cooked potatoes. We therefore undertook under RM:a-166 to develop cheap methods for producing a potato flour of good color from raw potatoes using direct heat dryers.

Since darkening on drying occurs largely on the surface, the overall color of the product could obviously be improved by drying potato slices instead of ground potatoes. We therefore attempted to dry raw potatoes sliced into uniform pieces in a single pass direct heat rotary dryer, using potatoes which had been stored at room temperature until their sugar content was approximately 1.2 percent MFB. It was found that the slices could be dried without sticking but only if the inlet gas temperature were kept at a maximum of 550° F. With potatoes which had been kept in cold storage until their sugar content was approximately 6.5 percent MFB, the maximum allowable inlet temperature was only 400° F.

In order to increase the permissible inlet gas temperature, thereby increasing the thermal efficiency and capacity of the dryer, studies were made coating sliced potatoes with a coarse potato meal to prevent sticking. This idea was an adaptation of German methods used during World War II. Using the coating meal, inlet gas temperature as high as 750° F. could be used without the slices sticking. However, when the same meal was repeatedly recycled, it progressively darkened, thereby impairing the color of the flour. Further studies are in progress.

Natural Freeze Drying of Potatoes. Natural freeze drying of potatoes has been carried out successfully in North Dakota and upper New York State during recent years. We initiated experiments of this nature in cooperation with the Maine Experiment Station at Presque Isle, Maine, on February 1, and jointly with the Cornell Experiment Station at Riverhead, Long Island, on February 21.

Potatoes were spread on the ground in various depths ranging from a few inches to one foot. They were also stored in horizontal and vertical open structures above the ground. It is hoped that conditions will be favorable for partial dehydration of these potatoes as a result of alternate freezing and thawing, followed by final drying out in the spring. Additional potatoes were put out in northern Maine in April when the season of alternate freezing and thawing normally is in progress. These experiments are to be followed up with observations and analytical determinations. If acceptable products are formed, feeding trials will be made.

Freeze dehydration of potatoes has been carried out in the laboratory by freezing potatoes each night for two weeks at -15 to -20° C. (near 0°F.) and allowing them to thaw in the daytime. Most of the juice is lost in the

first few thaws. After several cycles of freezing and thawing, it is apparently necessary to maintain the potatoes at ordinary temperature if further drying is to proceed with any rapidity. Some fermentation occurs. This may inhibit detrimental bacterial and fungal activities. Gases of fermentation seem to aid dehydration by forcing out juice. Although the laboratory experiment involved only a few pounds of potatoes, not one decayed.

Work Project No. RM:a-200 - Utilization of wool grease and other wool by-products

Studies on the fractionation of crude wool grease by molecular distillation have been completed. The use of molecular distillation as a means of separating wool grease into fractions having increased utilization does not appear very promising. Although more than one-third of the grease was recovered in a fraction which is partly liquid at room temperature no worthwhile concentration of desirable constituents appeared to be accomplished. Moreover, the sulfur present in the grease made such fractionation difficult.

The Wool Division of the Production and Marketing Administration has been endeavoring for some time to develop scourable sheep-branding paint, that is, a paint which would provide identifying marks when applied to the fleece and be permanent under range conditions but which would be removed from the fleece by the scouring operation which removes the grease. They have developed a paint consisting of wool grease, a grease solvent and a pigment which has been satisfactory in every way except that in cool weather it becomes too stiff to apply. Our assistance in the solution of this problem was requested. We felt that this defect might be obviated by removing some of the high melting compounds from wool grease by solvent fractionation. This was accomplished by dissolving crude wool grease in 5 volumes of a solvent such as isopropyl alcohol or methyl ethyl ketone at 60° C. and slowly cooling to about 5° C. The precipitate which formed was removed by filtration and the solvent was distilled from the filtrate, leaving a residue which is fluid at room temperature. This residue may be from 55 percent to 80 percent of the original grease. This material shows remarkably little change in viscosity which change in temperature.

About 20 pounds of this fraction have been prepared and submitted to the Wool Division of the Production and Marketing Administration for field evaluation as the base for scourable branding paints.

Work Project No. RM:a-307 - Conversion of vegetable and animal by-products, surpluses and wastes into new feedstuffs and the establishment of their feeding values

Seabrook Farming Corporation Contract. In December the Seabrook Corporation submitted a final report on the work accomplished during the 1949 season. This report assumed the character of an analysis of the changes which would be necessary for operation of the plant during the 1950 growing season. It was submitted to company officials to acquaint them with financial obligations if the waste dehydration program for the 1950 season was carried out on a production basis as compared with an experimental basis.



Two members of the Seabrook staff closely concerned with the program have conferred with staff members at this laboratory recently and although they were unable to speak for their company officials in regard to the program for this season it appears certain that an experimental production schedule will be necessary for at least part of the season before they are in a position to go into large scale production. Because of conveying and screening difficulties encountered during the past season a number of changes have been essential and these will be made within the next two months so that the first major crop (pea vines) can be handled in a more satisfactory manner than was possible last year.

One of the major considerations in the production of dehydrated vegetable wastes is their utilization by the feed industry. During the winter months the broccoli and spinach meals produced in the fall were sold to a local feed firm at a price considerably below their true value. This was done on the basis that they were experimental products and would not represent the future cost of similar leaf meals. One of the larger feed firms showed great interest in the high carotene content still present after several months of storage. This firm would be particularly interested if larger amounts of high quality meal were available.

In January a conference was held with staff members of the Division of Animal Husbandry, Bureau of Animal Industry, Beltsville Research Center, and plans were made for the testing of certain of the vegetable meals as stock and poultry feeds.

Processing Field and Cannery Wastes. The Heil two-pass rotary direct fired dryer and accessory equipment have been installed in a new fireproof dryer building. This equipment includes wet and dry screeners, hammer mill, forage cutter, continuous rotary press, mixer conveyor, and various conveying and metering equipment to form an integrated pilot plant. The plant will be used in the 1950 season on a wide variety of vegetable field and cannery wastes as well as potatoes.

Work Project No. RM:a-506 - Production of amino acids from milk proteins

Laboratory work on this project was initiated in October 1949. A study of the effect of acid concentration, protein concentration and temperature upon acid hydrolysis of casein was carried out first. It was found that a slight excess of hydrochloric acid above that necessary to produce amino acid hydrochlorides of the resultant amino acids produced complete hydrolysis in 4 hours in an autoclave at 15 pounds steam pressure. A study of the direct esterolysis of casein is now being carried out.

A number of butyl esters of amino acids have been synthesized and purified to determine their physical constants and stability under distillation processes.

Work Project No. RM:a-556 - Expanded research on fats and oils

This project has served to permit expansion of the work on (1) the oxidation of unsaturated fat acids, (2) the preparation of polymerizable

derivatives of animal fats, (3) glyceride composition studies, and (4) the preparation of soaps and detergents.

Studies of the oxidation of methyl oleate at 35° C and 70° C. in the presence of ultraviolet light have been completed. It has been found by infra-red examination that a cis-trans isomerization, apparently induced by oxygen, occurs.

Studies of the polymerization of the vinyl esters of the long-chain fatty acids and their copolymerization with other industrially important monomers have been continued and expanded. A technique for bead, pearl and dispersion polymerization has been developed.

While study of the physical properties of these polymers has not been completed, results to date are so encouraging that relatively large laboratory quantities of vinyl esters are required. In this connection arrangements have been made with General Aniline and Film Corporation to prepare, at no expense to the Government, about 30 pounds of crude vinyl laurate, vinyl palmitate and vinyl stearate. Availability of these esters will expedite our work considerably and at the same time it can be concluded that this company regards the work as being very promising.

Work has been re-opened on the investigation of glyceride structures and compositions of a number of common animal fats with the special view of developing a practical process for separating glyceride fractions that have potential application as special purpose fats. Solvent ratios as low as 5:1 have been used on several animal fats with considerable fractionation being obtained. It is anticipated that fractionated fats of inedible grade will be evaluated extensively for use in hot dip tin plating.



## NORTHERN REGIONAL RESEARCH LABORATORY

R. T. Milner, Director

## AGRICULTURAL RESIDUES UTILIZATION INVESTIGATIONS

Yield of Pentose Sugar from Corncobs Increased

Large-scale laboratory experiments under the Synthetic Liquid Fuels Project have demonstrated that the rate of conversion of the pentosans to pentose sugar during the first phase of the saccharification process can be increased by 15 percent when the countercurrent atmospheric hydrolyzation procedure which has heretofore been used is replaced by a concurrent procedure under elevated pressure. The yield of pentose sugar is greater because of a very noticeable decrease in the quantity of unattacked pentosans in the residues. Through this change, the raw material cost per pound of pentose sugar in the hydrolyzate can be lowered by one third.

Fermentation of Pentosan Hydrolyzate Liquors

Pilot-plant-scale investigations at the Northern Laboratory indicate that satisfactory yields of butanol, acetone, and ethanol can be obtained when the butanol fermentation is conducted on a mixture of 75-percent pentosan hydrolyzate and 25-percent corn. It was also found that in addition to other factors, when the copper content of the hydrolyzates is above certain levels the sugars are very difficult to ferment. Based on this observation, the cause for the appearance of copper in the hydrolyzates as well as its fluctuations was established. A simple and very economical procedure has been developed to decopperize hydrolyzates to a point where the remaining copper content can be considered negligible. These hydrolyzates, so treated, have given greatly improved fermentation results.

A limited number of samples of pressure-cooked pentosan hydrolyzates prepared in bronze cookers on a laboratory scale have been tested for fermentability. Results of fermentation trials in various media indicate that the fermentation characteristic of pressure-cooked hydrolyzates is substantially different from that of atmospheric pressure-cooked pentosan hydrolyzates. The pressure-cooked hydrolyzates thus far have been more difficult to ferment.

Composition of Corncobs

The results of a partially-completed study show that the pentosan content of corncobs from hybrid corn is higher than that of cobs from open-pollinated varieties. The pentosan content of open-pollinated varieties was 35 percent or less, while that of most hybrids was about 40 percent. One hybrid variety contained 45 percent of pentosans. The lignin content of most hybrids is around 12 percent, compared with 18 to 20 percent for open-pollinated varieties. However, one hybrid analyzed about 23 percent. The results of this investigation indicate the

possibility of developing cobs, by genetic studies, in which pentosans, lignin, or cellulose predominate. High pentosan content is extremely important if cobs are to be used for furfural manufacture. Lignin is important in relation to feeding.

#### Hydrogenation and Dehydrogenation Products from Furfural

As a result of continued studies on selective hydrogenation of furfural-ketone condensation products to furyl alkanols, methods have been found for separating these compounds from the spirofurans produced as side reaction products in amounts of 10 to 15 percent. The spirofuran produced in the selective hydrogenation of furfural-acetone has been proved to be 1,9-dioxo-2-methyl-5-spiro-nonane. The mechanism of spiroic formation involves the furylalkanol as an intermediate. Hydrogenation of furfuralacetone using a nickel-on-kieselguhr catalyst gave yields of 38 percent of the spirononane. This fundamental study may prove to be of industrial importance.

#### Structure and Synthesis of Gymnosperm Lignin

In order to prove that Alfred Russell's proposed "synthetic lignin," which was discussed in last year's report, really conforms to lignin as found in plants, it is necessary to show that the degradation products and other reactions of this lignin confirm to those of the various "lignins" studied by others. This work has been pursued along three lines: (1) The conditions of producing the Russell lignin from vanillin acetate by the Fries rearrangement have been carefully explored so as to produce consistent yields of a product which gives the elementary analysis that corresponds to the proposed formula for synthetic lignin. This work has been successful. (2) The product so obtained has been subjected to various oxidation reactions with the result that vanillin, vanillin-5-carbonyl acid, vanillic acid, and veratric acid have been isolated and identified, all of which have been obtained from natural lignins by other investigators. In all cases, however, yields were lower than those obtained by other investigators. (3) It seemed necessary, therefore, to synthesize the monomer of this synthetic lignin which Russell proposed to be 2-hydroxy-3-methoxy-5-formylacetophenone. Starting with ortho-vanillin, a 5-step synthesis of the monomer has proved successful. About 100 grams of this product have been prepared, purified, and its constitution established. Work using this product to produce the polymer called lignin by Russell is under way.

#### Mechano-Chemical Process Developed for Pulping Crop Residues

Experiments on the wet chopping of straw in preparation for cooking it with chemicals under increased pressure to produce paper pulp led to a simplified, economical pulping process that involves simultaneous mechanical and chemical action, eliminates the need for pressure-cooking equipment, and greatly reduces the time required for pulping. This mechano-chemical process, which utilizes the kind of commercial machine that is used industrially for disintegrating and



hydrating old newspapers as a step in producing deinked paper pulp, has promising possibilities for producing both strawboard pulp and bleachable fine pulp. This new process has proved successful on an industrial scale and industry is greatly interested in it.

### 9-Point Corrugating Board Improved

The cooperative research program undertaken by the Laboratory with industrial companies to improve the quality of strawboard has been most successful. Two years ago corrugating strawboard was proving inferior, particularly in rigidity, to board made from pulpwood by either the semi-chemical kraft process or the neutral sulfite process. Now that most of the mills have applied the results of the Laboratory's research, strawboard excels competitive wood products in stiffness.

The following specific accomplishments resulted from this program: (1) Improved refining conditions were developed whereby the cooked pulp could be processed in such a way as to develop its full strength properties on the paper machine. (2) It was found that in cooking procedures adapted to present mill rotary digesters, the use of lime, which has been the sole or major chemical cooking agent during the last 100 years, produced a pulp inferior in quality to that where other caustic chemicals were used. Modified cooking procedures, not employing lime, have been developed and applied industrially.

### Fine Straw Pulp

The Laboratory's neutral sulfite process for producing fine paper pulp is being more widely adopted in Europe. A new straw mill is being built in Paris, France, to use this process. Italian mills are adopting on an increasing scale the procedures and operating conditions developed by the Laboratory. A mill in Turkey is producing fine paper pulp from straw using the neutral sulfite process with caustic soda, since soda ash is not available at low cost. A number of the larger pulp and paper companies in the United States have expressed an interest in making mill runs of 10 or 20 tons of pulp, using this process.

### Soft-Grit Blasting

The use of grits from corncobs, pecan, English and black walnut shells, and apricot and peach pits as soft-grit blasting materials, which was pioneered by the Laboratory, is increasing. The General Motors Corporation is distributing information on corncob grits among its various agencies and distributors. The Tinker Air Force Base in Oklahoma is distributing similar information on pecan and black walnut shell grits. Corncob grits are now used in finishing parts for the hydramatic and similar automotive driving mechanisms. However, their newest and largest potential use is in low-pressure blasting to clean electric motors and generators, supplanting a solvent process which was time-consuming, costly, and hazardous. The Pangborn Corporation is now manufacturing an air blasting unit designed specifically for using soft grits from agricultural residues.

## CORN, WHEAT, AND OTHER CEREAL CROPS UTILIZATION INVESTIGATIONS

### Fundamental Studies on Starch Granule Structure

A better understanding of many problems in starch processing is being sought from knowledge of the behavior of starch in water absorption. Measurements have been made of the variation of water sorptive capacity as a function of physical treatment and chemical modification of corn and other starches, and of the relationships between water sorption and the properties of surface area, specific gravity, and swelling.

Starch granules are not nitrogen permeable as shown by surface area measurements of starches at low temperatures by gas adsorption techniques. On the other hand, water vapor is readily absorbed, and each type of starch exhibits upon water sorption a characteristic swelling behavior. Tapioca and waxy maize starches which find similar utilization show similar swelling for the amount of water absorbed, and differ from corn and potato starches in exhibiting greater swelling. The course of swelling upon water absorption is interpreted to be controlled by the unique organization of each starch. The similarity in swelling of the tapioca and waxy maize starches shows the importance of large-scale organization in obscuring molecular differences such as amylose-amylopectin ratio. It was also found that water sorption capacity is usually increased for starches in the initial stages of chemical modification, and this was attributed to loosened granule structure despite the absence of granule damage.

### Starch Sponge as a Hemostatic Agent

Much progress has been made during the past year on the use of starch sponge as a hemostatic agent. Experimental work on limits of starch concentration and freezing temperatures for the production of surgical sponge from corn, wheat, and potato starches has been completed. Publication by medical workers of clinical findings on the successful applications of starch sponge in cases involving hemorrhage has greatly stimulated interest and active study on this development. Dissemination of available information on the production, use, and value of starch sponge to the leading pharmaceutical firms in the country has further increased practical studies. Willingness of those groups to engage in practical tests will facilitate greatly the evaluation of starch sponge production. Varied clinical studies now being conducted by several drug companies are expected to lead to the early commercial production of starch sponge. Over one million pounds of starch per year may be required to supply this market outlet.

### Periodate Oxidation of Carbohydrates

In 1946, this Laboratory reported the discovery of a new glucosan, D-glucosan  $\langle 1,4 \rangle \beta \langle 1,6 \rangle$  which, although it was not oxidized by periodic acid, did contain adjacent hydroxyl groups. This evidence led us to question the validity of previously assigned carbohydrate



structures based only on oxidative reactions. The compound, D-galactosan  $\langle 1,5 \rangle \beta \langle 1,3 \rangle$ , described by Hann and Hudson was therefore open to question regarding the anhydric structure inferred from its behavior with periodic acid. We have now established that their compound has the structure D-galactosan  $\langle 1,4 \rangle \beta \langle 1,6 \rangle$ , thus providing another example of the anomalous behavior of carbohydrates having adjacent hydroxyl groups. Evidently gross spatial structure exerts considerable influence on the reactivity of adjacent hydroxyl groups. Care must be exercised, therefore, in interpreting evidence obtained with periodic acid reactions.

#### Improved Techniques for the Study of Amylose Hydrolysis by Malt Alpha-Amylase

Improved methods have been developed for the hydrolysis of amylose, the linear-molecule fraction of starch, with malt alpha-amylase and for the analytical study of the resulting products. A hydrolysis temperature of 70° C. makes possible the use of amylose concentrations as high as 4 percent without danger of retrogradation. In addition, this high temperature, together with the presence of calcium chloride added to stabilize the alpha-amylase, minimizes the possibility of interference by traces of beta-amylase.

The products of hydrolysis obtained at the achroic point under the conditions used have been fractionated both by alcohol precipitation and paper chromatography. Glucose is present in small quantities in the hydrolysis mixture. Alcohol precipitation appeared to yield a uniform fraction having about 7 glucose units per molecule. Paper chromatography demonstrated clearly that approximately 9 different oligosaccharides were present in this fraction. It is tentatively assumed that they constitute the homologous series of 1,4-linked glucose polymers which range in size from the disaccharide maltose (present in traces) through the 10-glucose unit dextrin.

The application of these findings and techniques to the mode of action of alpha-amylase on the amylose fraction of starch is being studied further.

#### Synthesis of D-Glucuronic Acid

A practical process of synthesis of D-glucuronic acid from dextrose has been perfected during the last year. This acid has long been known to be a compound of considerable physiological significance because of its ability to remove a number of toxic materials from the body. Glucuronic acid, or its lactone, has received much attention recently because of favorable clinical results in the treatment of a number of types of rheumatic arthritis. The value of the use of glucuronic acid in sulfa-drug therapy has also been indicated.

### Synthetic Detergents Derived from Dextrose

A series of sulfated gluconamides has been synthesized from gluconic acid and fatty acid amines. These compounds, prepared by condensation of two raw materials derived from farm commodities, has shown excellent detergent properties.

Soil-removing ability of the new synthetic detergents has been determined by the cooperative assistance of the Bureau of Human Nutrition and Home Economics. Data show that soil-removal efficiency increases with the length of the alkyl straight chain radical until about a 14-carbon-length is reached. N-Tetradecyl-D-gluconamide sodium sesquisulphate has optimum surface-active properties and gives a maximum soap index considerably higher than those usually obtained with commercial anionic detergents. Increasing the length of the alkyl chain to 16 and 18 carbons gave less soluble products, unless more highly sulfated, and the surface-active properties were somewhat poorer than those of the N-tetradecyl compound. Lower solubility of sulfates with a longer alkyl chain is probably a factor in their reduced soil-removal efficiency.

Preliminary tests have shown that surface-active properties of mixtures of N-tetradecyl-D-gluconamide sodium sesquisulfate with sodium sulfate, carboxymethylcellulose, or sodium triphosphate, in which the detergent comprises 34 percent by weight, do not vary markedly from those of the detergent itself.

If large-scale production of fatty gluconamide sodium sulfates for detergent use appears economically feasible, a considerable volume of both fats and corn would be consumed.

### Production of Potassium Acid Saccharate

Methods have been developed which have reduced the cost of chemicals for production of potassium acid saccharate from dextrose. The requirement of a large excess of nitric acid to oxidize the dextrose, reported last year, has been lowered. Dextrose has been oxidized with as little as 2 1/2 moles of nitric acid to 1 mole of the carbohydrate. For a single batch experiment, a 3 to 1 molal ratio yielded 50 pounds of potassium acid saccharate from 100 pounds of dextrose, representing a satisfactory yield for this reaction. A reduction in the cost of chemicals is attained by neutralizing the oxidation liquor first to a pH of 3.8, obtaining thereby about 60 percent of the potassium acid saccharate present. The remainder of the salt can be recovered by neutralizing further to a pH of 9, then reacidifying to a pH of 3.4. Low temperatures during these neutralizations are necessary to obtain white crystals and light-colored solutions. It was found that a reduction in processing and standing time aided in holding color formation to a minimum.

The potassium acid saccharate and the solution remaining after its removal are sequestrants. It was determined that liquors produced by



oxidation of dextrose could be neutralized with sodium hydroxide to make a liquid product with good sequestering power. Likewise hydrol, a byproduct of dextrose manufacture, can be oxidized with a small amount of nitric acid (1 to 1 molar ratio) and neutralized with sodium hydroxide to make a cheap sequestering agent.

#### Production and Utilization of Fungal Amylase

A synthetic medium consisting of starch and inorganic salts has been developed for the satisfactory production in the laboratory of fungal maltase in submerged, aerated cultures of Aspergillus niger NRRL 330. Yields of more than 30 maltase units per ml. of culture have been obtained. The culture filtrate from the synthetic medium used in maltase production was vacuum concentrated at 38° C. to about one-tenth the original volume without loss of enzymatic potency. Dry preparations, approximately 50,000 maltase units per gram, have been obtained by further purification of the vacuum concentrate through electrodialysis and ethanol precipitation procedures.

Aspergillus niger NRRL 330 produces culture filtrates which hydrolyze isomaltose to a greater degree than filtrates of Aspergillus niger NRRL 337 reported last year. This ability to break down isomaltose is of considerable interest since the linkage between the two glucose residues in that sugar is of the  $\alpha$ -1,6 type. This type of linkage occurs at points of ramification of the amylopectin molecule and is resistant to action of the common amylolytic enzymes.

Experiments have been conducted in the pilot plant to determine the suitability of various grains other than sound corn for use in the production of ethyl alcohol with fungal amylase as the converting agent. Moldy corn, hard and soft sound wheat, and sound grain sorghum have been utilized in these studies. The enzymatic liquor used in all experiments was produced from thin stillage with a solids content of 4 percent to which had been added 5 percent of corn, wheat, or sorghum meal. In studies using moldy corn, satisfactory fungal amylase liquors were produced when the medium was aerated at a rate of 1/4 volume of air per minute per volume of liquor. For the other grains, it was necessary to increase the rate of aeration to 1/2 volume of air per minute per volume of medium.

For all grains that have been tested thus far, yields of alcohol obtained with fungal amylase as the converting agent were at least equal to those obtained by the use of malt. No operating difficulties were encountered in any of the tests.

#### Production of Citric Acid from Glucose

Procedures have been established whereby good yields of citric acid can be obtained from untreated commercial grades of glucose in submerged culture. The main problem is to speed up the fermentation. A number of nutritional and environmental factors influence mold growth and acid production, and the problem of evaluating these factors singly and in combination becomes very complicated.

Recent work has emphasized the relation between temperature and nitrogen supply as these factors affect both growth and acid production. The amount of aeration appears to be closely correlated with the amount of mycelial growth.

Results obtained suggest that some choice can be made between temperature and nitrogen concentration, especially in a synthetic medium. The fermentation rate in 7-day old cultures has been nearly doubled during the past year by effecting changes in type of shaker, incubation temperature and nitrogen supply. Good yields have been obtained by submerged culture in small stainless steel vats. Further work is required, however, to correlate and evaluate the various factors known to influence this fermentation.

#### Production of Sodium Gluconate

Pilot-plant studies were conducted on the development of a process for the preparation of sodium gluconate by fermentation of glucose with Aspergillus niger NRRL 3. The work was conducted in accordance with terms of an informal memorandum of understanding between the Diversey Corporation, Chicago, Illinois, and this Laboratory. This corporation is interested in using the material as a sequestering agent in washing compounds of high alkalinity.

Deep vat fermentations in 200-gallon batches were conducted, using a medium containing approximately 20 percent sugar and appropriate nutrients. During fermentation, the medium was aerated at a rate of 1 volume of air per minute per volume of medium. The pH of the medium was controlled at 6.0-6.5 by the automatic addition of sodium hydroxide to neutralize the gluconic acid as it was produced, thereby forming sodium gluconate.

Sodium gluconate was recovered by filtering the beer, concentrating the filtrate to 40 percent solids in an evaporator, and then drying the sirup on a drum drier. Approximately 2,800 pounds of material were produced and submitted to the Diversey Corporation for consumer tests, which are now in progress.

A preliminary cost estimate based on a plant producing 3 million pounds of product annually shows the cost to be 9.6 cents per pound.

#### Polymyxin--An Antibiotic

Further research on the antibiotic, polymyxin, reported 2 years ago, has disclosed certain unique properties. Crude, carbon-adsorbed polymyxin D, when fed to normal poultry in levels of 0.5 to 1.0 percent of the diet, induced the following changes in the birds: (1) The normal, predominantly gram-negative, bacterial intestinal flora is replaced by a gram-positive flora, and (2) a marked increase in weight (17 percent) occurred in the birds which received 1.0 percent supplement of carbon-adsorbed polymyxin to the diet as compared with



the controls which received no polymyxin. Although feeding of polymyxin as a routine constituent of the normal poultry diet may not be feasible, there is a good possibility of using it in medicated diets for limited periods to minimize or control infections of the alimentary tract.

Tests are now in progress at the Carnation Laboratories in Milwaukee, Wisconsin, to determine the effect of polymyxin in a medicated feed on the course of coccidiosis in experimentally-infected chickens. Similar tests have been undertaken at the Department of Veterinary Pathology, University of Illinois, Urbana, Illinois.

#### Manual of the Penicillia

The "Manual of the Penicillia" by Raper and Thom, which was prepared under the joint sponsorship and support of the Bureau of Agricultural and Industrial Chemistry and The National Science Fund, was published in August 1949. This represents, first and foremost, a treatise on the taxonomy and relationships of the Penicillia, a genus of molds important to agriculture and industry. It brings together all literature relative to the biochemical, physiological, and pathological activities of these molds. One hundred and forty-two species and varieties are recognized. Descriptions and illustrations of the recognized species, together with treatments of the synonymy of species in the literature should greatly facilitate the identification of molds belonging to this unusually abundant and difficult genus.

#### Cyanoethylation of $\alpha$ -Amino Acids

Cyano- and dicyanoethyl derivatives of amino acids and amino acid mixtures have been prepared. These derivatives, particularly the dicyanoethyl, are unstable to high temperatures, losing acrylonitrile. When these cyano derivatives are converted to esters, they are also unstable to heat, losing alkyl acrylate. The mono-cyanoethyl derivatives and their esters can be stabilized by converting the amino group to the amide. Properties of these materials have been determined, and additional work on their derivatives to ascertain their utility is now being undertaken.

#### Study of Alcohol Motor Fuels in Automotive Engines

Following preliminary trials, four 200-hour tests on engine wear were run on full-scale stationary engines using (1) gasoline and (2) a 25 percent denatured alcohol blend with gasoline. Average wear data, obtained from a total of 64 indentations in the cylinder walls made at four levels of piston travel, gave .0005 inch of wear for the gasoline and .0003 inch for the 25 percent blend, indicating that the alcohol blend produced less wear, and that the denaturant used, ST-115, was not harmful under test conditions. Results were very consistent in each of the two sets of runs.

Three 10,000-mile runs with a 25-percent denatured alcohol blend with gasoline showed a fluffy carbon deposit in the combustion chamber in contrast with a harder carbon deposit formed with gasoline alone.

## SOYBEANS AND OTHER OILSEED CROPS UTILIZATION INVESTIGATIONS

Contributing Causes to Flavor Instability of Soybean Oil

In previous reports, the beneficial effects of small amounts of citric acid added to soybean oil during deodorization have been reported. The effect of citric acid was attributed by the Germans to removal of traces of lecithin, but it was found at the Northern Laboratory that citric acid actually serves as a metal scavenger, especially for iron. Other metal inactivators of equal function have also been discovered and reported. During the past year a method has been developed for measuring the very minute quantities of iron and copper which are so harmful to flavor stability of soybean oil. The analytical procedure involves ashing of a large oil sample and spectrographic analysis of the ash, after combining with a small quantity of buffer to stabilize the line intensities and introducing known amounts of beryllium as a standard. With this analytical method it was found that the presence of over 0.1 part per million (p.p.m.) of iron, or 0.02 p.p.m. of copper, definitely reduced the flavor stability of the oil after accelerated aging at 60° C., and after storage for several weeks at room temperature. Addition of iron above these levels gave proportional decreases in stability while the addition of small amounts of citric acid during deodorization counteracted the effect of the metal.

For comparative purposes, cottonseed oil was also studied. As much as 0.3 p.p.m. of iron added to cottonseed oil did not affect its flavor stability. Since cottonseed and soybean oils differ in composition largely by the presence of linolenic acid in the latter, this difference emphasized the role of this triply-unsaturated acid, as well as iron, in flavor reversion. Investigation and confirmation of the effect of linolenic acid was undertaken and reported on under a Research and Marketing Project.

Commercial Soybean Oils

With iron singled out as a chief offender in the laboratory studies and with citric acid and other metal inactivators as possible remedies, the commercial application of this knowledge became of importance. Soybean oil is removed from the beans commercially in iron equipment either by solvent extraction, continuous screw pressing, or hydraulic presses. Soybeans were carefully solvent-extracted in all-glass apparatus in the laboratory and found to contain approximately 0.8 p.p.m. of iron so that any additional iron acquired during processing might have a very serious effect on flavor stability. Four commercial processing plants were visited. Samples were obtained at various processing stages and also as the oil passed on through the alkali refining, bleaching, and deodorizing stages needed to produce a salad oil. As could be expected, iron content rose as a result of processing but was markedly reduced during alkali refining. In some of the plants, iron was reintroduced during deodorization. In spite of high iron content, however, some of the sampled oils proved exceptionally stable



which indicated clearly that a metal inactivator had been used during deodorization. Information could not be obtained from some of the plants furnishing the samples but, where available, the use or non-use of metal inactivators confirmed laboratory findings. These studies also emphasized the great importance of the deodorization process on the flavor of fresh oil. In several cases it was found that a laboratory deodorization produced an oil much superior to the same oil when plant deodorized.

This work clearly indicates that iron is introduced in processing soybeans for oil, that unless removed or inactivated, it plays an important role in subsequent flavor stability of the oil, and that deodorization is of great importance in governing the flavor of fresh soybean salad oil.

#### Flavor Stability of Oils Other Than Soybean

With a taste panel trained for research on the flavor of soybean oil it is easily possible to evaluate, for comparative purposes, other oils of present or future interest. Oils from safflower, mustard, sorghum, and sunflower have been tasted as refined, bleached, and deodorized salad oils. As fresh oils, safflower was judged the best, with mustard seed oil a close second. None of these oils was outstanding in flavor stability. The raffinate (low iodine number fraction) produced by extraction of soybean oil with furfural had excellent flavor stability. It rapidly became rancid, however, unless protected by anti-oxidants since most of the natural anti-oxidants were removed by the furfural.

#### Characteristics of Esters and Acids

The separation of fatty acids by distillation of their methyl esters is a well-known method of obtaining pure samples of these esters. Distillation is employed commercially to produce fractions of enhanced commercial value. Exact vapor pressures have been determined for methyl esters of acids containing from 8 to 18 carbon atoms in order to calculate the design and usefulness of fractionating columns for separating these acids.

#### Refining Loss and Sludge in Soybean Oil

When tank cars of crude soybean oil are shipped from processor to refiner, there is frequently deposited enroute a layer of sludge, or settlings, in the bottom of the car. This material arises largely from the phosphatides and other non-glyceride materials in the crude oil. Refiners dislike receiving tank cars containing sludge, and present trading rules provide for price adjustments when the amount of sludge exceeds 150 pounds (a tank contains about 61,000 pounds oil). Since the price of oil is adjusted according to refining loss measured on a sample taken when the tank car is loaded, the relationship between refining loss and sludge is an important one, both with respect to the price of oil and good will between buyer and seller.

Laboratory tests were undertaken at the request of the Technical Committee of the National Soybean Processors Association. To date, preliminary tests indicate that there is a decrease in refining loss as sludge deposits and that the present penalties, on both sludge and refining loss, may need adjustment to be fair to both buyer and seller. Further tests are under way.

#### New Method of Locating the Double Bond in Isomers of Oleic Acid

It has been claimed that the presence of vaccenic acid (an oleic acid isomer with the double bond between carbons 11 and 12) in summer butter is responsible for increased nutritive value of this fat. In attempting to synthesize this acid for study, the question of analyzing and distinguishing the various oleic isomers was raised. It has been found that conversion of these unsaturated acids to dihydroxy stearic acids was easy. X-ray diffraction patterns of the acids were then made and elaidic (9-10) and vaccenic (11-12) acids, both of which are solids, showed differences in intensities which could be used to identify the presence of the separate acids. This method will be studied further as a useful tool for distinguishing among these isomers.

#### Cooperative Work on Improvement of Nutritional Value of Soybean Oil Meal

At the request of several large commercial companies selling mixed feed, the Northern Laboratory has organized a cooperative group from industry, state agricultural experiment stations, and other Government agencies to study possible improvements in the nutritive properties of soybean oil meal. To serve this group, the Laboratory processed 4,300 pounds of soybeans in its pilot plant for continuous solvent extraction to produce 2,575 pounds of oil meal. This meal was divided into 10 lots, 5 of which were heated (toasted) at atmospheric pressure for varying times and 5 were heated for 15 minutes to varying temperatures (as fixed by definite steam pressures). These treatments and the apparatus used approximated commercial practice and were designed to give meals subjected to conditions both milder and more severe than are normally met in industrial processing. Feeding tests on rats and chickens were made by the collaborators to correlate, if possible, the nutritive value of the meal with the heat treatment. Results on feeding tests by the various collaborators, however, were most conflicting. Further study is needed to determine if the difficulties encountered arose in the feeding tests or in the preparation of the test meals. Individual efforts are now being made to explain the discrepancies found on feeding trials in different laboratories before continuing further collaborative work.

#### Pilot-Plant Production of Gelable Soybean Protein--Gelsol

The pilot plant mentioned in last year's report has been constructed and operated by a nearby soybean processor in close cooperation and with the assistance of Laboratory personnel. Many difficulties



were encountered. In removing alcohol from soybean flakes, excessive denaturation of the protein occurred which so lowered its water solubility that the yield of Gelsoy was greatly reduced. In the several screening operations employed to separate solids and liquids, a large amount of fines was formed which carried through to the final product. This last difficulty was solved by the use of a centrifuge. Laboratory studies showed that either very low or very high concentrations of alcohol did not cause appreciable denaturation, but that a mixture of water and alcohol in equal amounts denatured the protein within a few minutes both at 30° and 75° C. These observations explained the failure of conventional desolventizing equipment and indicated flash drying was required for this process.

## RESEARCH AND MARKETING ACT PROJECTS

### New Structural and Chemical Studies of Grain Kernels

Studies on the histological structures of the entire corn kernel, now completed, have been recorded photomicrographically. These pictures record the types of tissues present, and show the structural relationships of importance from the standpoint of industrial utilization.

Histochemical studies of corn are still in progress. Cellulose has been found in cell walls throughout the kernel; hemicelluloses form a large part of the cell walls in the hull and occur in small quantities in the cell walls of the embryo and endosperm. Lignin was found only in the tip-cap region. The spermoderm and the closing layer of the hilar orifice, which together form a semi-permeable membrane around the corn kernel, were found to be composed of suberous or cork-like material. The knowledge of the chemical nature of these structures is basic to the treatment required to effect their separation in milling methods.

### Production of Riboflavin by *Ashbya Gossypii*

Laboratory and pilot-plant studies have been continued on a process for producing riboflavin by fermenting a medium consisting of glucose, corn steep liquor, and animal stick liquor with the yeast-like organism, *Ashbya gossypii*. Careful review and analysis in the laboratory employed in this fermentation have led to a stricter regimen involving maintenance of stock culture, preparation of the inoculum, and use of reduced amounts of inoculum. These measures substantially increased yields and gave greater reproducibility between fermentations.

Production of the vitamin by this process on a commercial scale is economically feasible. The possibility of further improving yields of riboflavin in large-scale fermentations exists in the fact that laboratory results are favorably affected by low concentrations of certain inorganic salts. Pilot-plant work has confirmed laboratory experiments in showing that yields can be increased by addition of sugar to the standard medium about 40 hr. after inoculation.

### Vitamin B<sub>12</sub> Synthesis by Microorganisms

A screening procedure was developed for surveying the Laboratory's Culture Collection for vitamin B<sub>12</sub>-producing microorganisms. With the assay method used, none of the 2,050 yeasts or 2,100 molds examined during the year was found to produce significant amounts of vitamin B<sub>12</sub>. However, some strains of the nearly 500 actinomycetes (*Streptomyces*) were found to synthesize varying amounts of this vitamin. A survey made last year of about 1,000 cultures of bacteria disclosed a small number of promising strains for the production of vitamin B<sub>12</sub>.

When *Flavobacterium devorans* NRRL B-54, one of the more promising organisms for B<sub>12</sub> production, is grown under ideal conditions on a medium composed of glucose, soybean meal, corn steep liquor, and mineral salts, the vitamin is produced in amounts sufficient to interest the fermentation industry. Studies are continuing to develop this process to give consistently high yields.

### Fungal Amylase Process for Plant-Scale Conversion of Sound Grains into Alcohol

To determine the practicability of the fungal amylase process developed at this Laboratory, and to obtain pertinent engineering data, the U. S. Department of Agriculture contracted the Grain Processing Corporation, Muscatine, Iowa, to make large-scale runs to produce alcohol from sound corn employing this process.

Plant tests made so far indicate that the preparation of good quality fungal amylase liquors on a large scale is practicable. It has been demonstrated that a mold agent of satisfactory enzyme potency can replace malt entirely in the mashing of sound corn with no disruption in subsequent plant operations or detrimental effect on the alcohol yield.

### Alcohol-Water Injection Studies on Internal Combustion Engines

Test results on three high-compression automobile engines (10:1, 8:1, and 8.25:1) show that two of the engines could be operated with present-day gasolines in combination with alcohol-water injection. The test engine with 10:1 compression ratio required the highest-grade premium gasoline in combination with injection for economical operation. There was very little difference in effectiveness between methanol- and ethanol-water mixtures; isopropanol was definitely less efficient.

Manifold distribution, the means by which an individual cylinder in a multi-cylinder engine receives its fuel, has received considerable attention since the high heats of vaporization of alcohol and water, introduced by injection, may cause unequal mixture distribution. A slightly different pattern and a wider variation were found in the manifold distribution of alcohol-water mixtures compared with that of gasoline. Both alcohol-water mixtures and gasoline are greatly affected by mechanical factors in the intake system.



## Linolenic Acid and the Flavor Stability of Soybean Oil

Soybean oil contains linolenic acid as one of its component fatty acids as contrasted with cottonseed oil which is free from linolenic. Since the flavor stability of cottonseed oil is superior to that of soybean oil, it has been postulated frequently that linolenic acid may be responsible for the undesirable flavors found in aged soybean oil. Direct evidence has been obtained to show that linolenic acid, when introduced into cottonseed oil, gave flavors on aging which were identified by the Laboratory's research taste panel as soybean oil flavors. To prepare the modified cottonseed oil for this study required considerable work and several attempts. It was necessary to prepare bland oils for all taste tests and this required a careful study of the catalysts used to introduce linolenic into cottonseed. Two samples of cottonseed oil were modified, one with linolenic acid, the other with linoleic acid (a normal component of cottonseed oil). These two oils were compared by the taste panel with samples of soybean and unmodified cottonseed oil. The taste panel reported, with statistical significance, that after aging, the cottonseed oil modified with linoleic acid was a cottonseed oil in flavor but that the cottonseed modified with linolenic acid was a soybean oil. This conclusion, that linolenic acid is (with metals such as iron or copper) an important factor in flavor "reversion," is also supported by the improvement found when a large part of the linolenic glycerides were removed by liquid-liquid extraction. Oils of superior flavor stability resulted when the linolenic acid content was lowered from 7-9 percent to between 2-4 percent.

## Isolation and Characterization of Flavor Principles

Under contract research at the University of Pittsburgh, continued progress has been made in identifying the compounds found in the distillate from "heat-reverted" soybean oil. Two aldehydes, decadienal and acetaldehyde, were isolated and identified. In a similar distillate from hardened soybean oil, dipropyl ketone and  $\alpha$ -heptenol have likewise been found. Other compounds are known to be present and are being studied.

## Composition of Soybean Phosphatides

Studies with the Craig countercurrent distribution apparatus and immiscible solvents have shown soybean phosphatides to consist of three main groups. The choline (lecithin) and ethanolamine (cephalin) fractions are alcohol soluble; whereas, the alcohol insoluble fraction consists of a complex mixture of inositides. Sugars usually accompany these, but there is still uncertainty as to whether the carbohydrates are merely carried along or are chemically attached. Galactose and fructose have been identified (after hydrolysis) by paper chromatography. The isolation and identification of the various components require a great deal of work. Frequently, the modification of an old method or devising of a new method of analysis is necessary. For comparative purposes, corn phosphatides were fractionated in the same manner as that for soybean phosphatides. The same components are present but in markedly different amounts.

AGRICULTURAL CHEMICAL RESEARCH DIVISION

L. F. Martin, Head

Research work of this division is largely conducted in strategically located field laboratories. Citrus fruit processing is studied at Winter Haven, Florida, and Weslaco, Texas; cucumber pickling at Raleigh, North Carolina, sugarcane processing at Houma, Louisiana, and tung oil and meal production at Bogalusa, Louisiana. Basic research on cane sugar and molasses and confectionery is carried on by the Division at the Southern Regional Research Laboratory in New Orleans.

SOUTHERN FRUITS AND VEGETABLES PROCESSING  
INVESTIGATIONS

Investigations of Texas and Florida Citrus

The investigations on the composition of lipids in citrus juices is fundamental work aimed at improvement in the quality of canned juices. If the composition and the changes that take place are known, it will be much easier to devise means of correcting undesirable changes.

The bacteriological work has been of assistance in establishing methods of quality control, particularly in routine plating and the evaluation of data obtained on coliform organisms. This product has enjoyed a phenomenal growth in the past 4 years and much of this has been due to the care taken in quality control including sanitation.

The investigations on the addition of citrus molasses to dried citrus pulp have shown that no trouble should be encountered with this product if it is properly prepared. This provides another way of utilizing citrus molasses, which has been a "distress" product, financially.

The Weslaco Laboratory has been advising local grapefruit and orange processors on problems and general types of equipment necessary for production of frozen concentrate. When concentrate plants are in actual operation, the data on Texas orange and grapefruit concentrate and various concentrated blends which are now in process of evaluation will prove of inestimable value to processors. Future studies will be aimed at keeping ahead of any likely problems.

The demonstration that the color of red grapefruit is stable under normal conditions of low temperature concentration and storage opens a market for cull pink and red grapefruit. These fruits were beginning to present a disposal problem, since the color is not stable in canned juice, and deteriorates to a muddy brown.



### New Knowledge of Enzymes an Aid to the Pickling Industry

The identity of the chemical agent (enzyme) responsible for the softening of salt-stock cucumbers is a research accomplishment of major potential importance to the pickling industry. Companies are now starting routine examination of their vat brines for the softening agent according to the procedure developed during the above study. This will permit them to have accurate information on the outcome of the cured material and will materially reduce losses previously suffered by not knowing which vats of material should be used, or which could be kept stored longer in brine.

Information on the origin of the chemical agents (enzymes) responsible for softening of brined cucumbers is also an important recent contribution. The softening enzyme has been found in high amounts in the seeds of several varieties of pickling cucumbers, as well as in the male flowers and pollinated female flowers. It has also been found in the large-sized cucumbers as they approach the ripe stage. These findings demonstrate that the cucumber that is brined may have an important bearing on the firmness of the cured salt-stock. This applies also to the brining of small cucumbers which still have the blossoms attached.

Another enzyme, called pectinesterase, has been shown to be present in practically all parts of the cucumber plant as well as the cucumber fruit. It assists the softening enzyme in destroying the pectin (cementing material) in the cucumber. Heretofore, neither of the two enzymes mentioned has been shown to be present in cucumbers, or the various parts of the cucumber plant. Such information is of vital importance in establishing a means of controlling the softening of salt-stock.

### CANE SUGAR, SIRUP, AND BYPRODUCTS INVESTIGATIONS

#### Clarification of Cane Juice and Recovery of Aconitic Acid Studied

Critical studies of juice clarification processes are being made in order to rationalize the chemical and physical principles that are involved. The findings obtained to date indicate that certain changes in the ordinary clarification procedure would be helpful, if adopted in reducing some of the trouble in the processing of Louisiana cane juices. Further, there is indication that calcium carbonate can profitably be used as an aid to juice clarification in the production of direct consumption sugars and fancy molasses but large-scale testing needs to be done before recommending it to the industry.

Molasses is a surplus commodity and its utilization is important to the welfare of both growers and processors of sugarcane. By ion-exchange it is possible to recover 95 percent or more of the aconitic

acid present and at the same time obtain a greatly improved molasses product which may find wider use. Solvent methods for recovery of aconitic acid are also being investigated.

#### Research for the Candy Industry

A safe molding medium has been developed for the candy industry. It has been demonstrated that replacing starch powder by a calcium carbonate-starch mixture will eliminate the explosion hazard in candy molding operations and at the same time provide a more flexible medium for control of moisture absorption and quality of the candy. Several insurance companies are interested and insurance savings might be considerable.

Many products such as butter creams and butter caramels disappear from the market in the summer because of their poor keeping qualities. The present effort to select suitable antioxidants for fat preservation in candies would be of great value in extending and reducing losses in marketing such candies.

#### TUNG NUTS INVESTIGATIONS

This project is under the direction of K. S. Markley, Head, Oil and Oilseed Division, Southern Regional Research Laboratory.

#### Tung Press Cake Dust an Explosive Hazard

Recent studies on various dusts collected at tung mills have shown the tung press cake dust to present a fairly high explosion hazard. Any of the dusts tested could cause fires or explosions. After the results of these tests were brought to the attention of mill managers, they made an effort to keep down dust concentrations in the mills by installing explosion-proof motors, switches, and other approved electrical equipment.

#### New Methods of Analysis Provided Tung Industry

The whole fruit methods of sampling and analyzing tung fruit developed by the Tung Oil Laboratory at Bogalusa, Louisiana have given the domestic tung oil industry reliable and accurate techniques for the analyses of tung fruit. These methods were used by the Commodity Credit Corporation in carrying out its 1947 Tung Oil Program and have been used successfully for several seasons by a number of commercial oil laboratories. As the tung fruit producers are paid for their crop on the basis of the oil content of the fruit, reliable and accurate methods of analyses were urgently needed to insure the growers full value for their crop.



Methods developed for the analysis of commercial tung hulls for oil content have been applied to a study of hulling efficiency and have yielded valuable information on oil losses in the hulling operations. This information will be useful to growers in determining whether it is more advantageous to haul the whole tung fruit to the mill (in which case the loss of oil in hulling is assumed by the mill), or to hull the fruit in the grove and then ship the hulled nuts to the mill for processing.

#### RESEARCH AND MARKETING ACT INVESTIGATIONS

##### New and Improved Uses of Citrus Products (RM:s-140)

The investigations under this project, which dealt with effect of pasteurizing conditions, are providing information on the exact temperatures required for pasteurization of citrus juices to obtain an optimum quality.

The development of a rapid and accurate test for pectinesterase will be of value in controlling quality in commercial canning.

DIVISION OF BIOLOGICALLY ACTIVE COMPOUNDS

Thomas D. Fontaine, Head

BASIC INVESTIGATIONS IN THE CHEMISTRY OF AGRICULTURAL PRODUCTS

PLANT DISEASE

Further Characterization of Tomatine and Tomatidine

Tomatine has been characterized as a glycoside comprising alkaloid and carbohydrate fractions. When tomatine is treated with acids the molecule splits into tomatidine and a carbohydrate fraction. A paper-partition chromatographic apparatus was developed with which a separation of the carbohydrate fraction into its component parts was achieved. Chemical tests applied to these sugars in comparison with known sugars as standards established their identity as xylose, glucose, and galactose. It was found that the tetrasaccharide which comprised the carbohydrate fraction was made up of two molecules of glucose and one each of galactose and xylose. Tomatidine has been further characterized by preparation of diacetyl and dihydro derivatives. Tomatine and tomatidine, supplied to the Pharmacology Division, Western Regional Research Laboratory, have been found to be relative non-toxic when administered orally to experimental animals.

PLANT-GROWTH REGULATORS

Radioactivity Aids Growth-regulator Research

The synthesis of a new plant-growth regulator, 2,4-dichloro-5-iodophenoxyacetic acid, was announced in last year's report. Twenty millicuries of radioactive iodine was used in synthesizing 18 grams and imparted a low degree of radioactivity to the compound and 11 derivatives (ammonium, ethylammonium, diethylammonium, triethanolamine, sodium, and calcium salts; an acid chloride; an amide; and methyl, isopropyl, and n-butyl esters). During the past year, in cooperation with the Bureau of Plant Industry, Soils, and Agricultural Engineering, these compounds, labeled with radioactivity, were applied to different plants, and their distribution in the plants was determined. They were found to accumulate primarily in the newest tissues at growing points.

The new regulator, 2,4-dichloro-5-iodophenoxyacetic acid, is closely related to the well-known weed killer, 2,4-dichlorophenoxyacetic acid and although its plant-growth regulating activity is only about 60 percent of the latter, due to the substitution of iodine for hydrogen in the benzene ring, the kind of plant response to it is quite similar. The esters and amide derived from the regulator containing radioactive iodine had greater regulating activity than their parent acid when tested on an equivalent-weight basis, whereas the salts had lower or no better activity than their parent acid. The growth-regulating activity of some esters approached but in no



case equalled that of 2,4-dichlorophenoxyacetic acid. Complete evaluation of the results obtained with such compounds should add to the present meager knowledge of the mechanism of action of plant-growth regulators.

#### New Synthetic Compounds Tested for Growth-regulating Properties

More than 150 new synthetic compounds were prepared as possible plant-growth regulators and are being tested in cooperation with the Bureau of Plant Industry, Soils, and Agricultural Engineering. These compounds include substituted amino acids, phenyl, phenoxy, pyrimidine, naphthalene, pyridine, quinoline, and mercapto compounds. An attempt to prepare one new plant-growth regulator, alpha-naphthalene butynoic acid, resulted in the synthesis of two isomers. The molecular configuration of each is being determined. One isomer has a melting point of 112°C. and is very active as a plant-growth regulator, whereas the other melts at 127°C. and is much less active. The activity of the very active isomer is somewhat similar to that of alpha-naphthalene acetic acid which promotes rooting and gall formation. This is the first aromatic-acetylenic acid to be tested as a plant-growth regulator, but other compounds of this general type are being synthesized.

#### INVESTIGATIONS CARRIED OUT UNDER THE RESEARCH AND MARKETING ACT

##### PRODUCTION OF PLANT-GROWTH REGULATING COMPOUNDS FROM AGRICULTURAL SOURCES; RM:a-272

#### Corn Pollen and Distillers Solubles

Two main sources of natural plant-growth regulators have been investigated, namely, corn pollen and distillers dried solubles (corn and barley). Fractionation of extracts obtained from corn pollen has resulted in the partial purification of the plant-growth regulators present. However, no evidence for the presence of indoleacetic acid has been obtained. Fractions obtained from distillers solubles have been purified to the extent that some show plant stimulatory activity at 1 to 50,000 dilution. There is chemical evidence for the presence of an indole compound in these fractions, and the ultraviolet absorption data are almost identical with those reported for an unidentified factor essential for the growth of animals, called Vitamin B<sub>13</sub>. If the plant-growth stimulator and vitamin B<sub>13</sub> are the same, it has been obtained in a higher degree of purity than previously reported. There is also a fraction in distillers solubles which kills plants, but not much success has been achieved in purifying this particular fraction. All fractions prepared by this Bureau are submitted to the Bureau of Plant Industry, Soils, and Agricultural Engineering for plant evaluation and application.

## PRODUCTION OF ANTIBIOTICS FROM AGRICULTURAL SOURCES; RM:a-145

### Survey of Various Plants and Plant Parts as Sources of Antibiotics

Extracts of plants and plant parts have been assayed for antibiotic activity. Of the one hundred and seventy five investigated, only a few plant extracts failed to exert some effect on the growth of at least one of the test microorganisms. Plants are, therefore, a potentially rich source of antibiotics, with a wide range of selectivity and specificity toward the bacteria, fungi, and yeasts which cause diseases in man. In many instances, these antibiotics occur in by-products of the agricultural and industrial processing industries, such as sweet potato, pea, peanut, and lima bean vines; grapefruit, orange, and lemon peel; turnip, spinach, beet, broccoli, and rhubarb leaf meals; cabbage, and carrot tops.

#### Antibiotics from the Sweet Potato Plant

Sweet potato vines, which are normally used as silage, have been found to contain highly active antifungal and antibacterial substances. It is perhaps significant that the edible tuber also contains these substances. Sweet potato vines, in a tonnage quantity, have been dried in a continuous drier and antibiotically active concentrates have been prepared from the dried product. Fractionation of these concentrates is proceeding rapidly. From an active water-soluble resinous fraction, a buff-colored crystalline appearing solid and a clear red-brown liquid, with a distinctively characteristic odor, have been obtained. The solid material exhibits selective activity toward Gram-negative (E. coli), and the liquid toward Gram-positive bacteria (especially Mycobacteria), and toward fungi. Toxicological and in vivo antibiotic activity will be determined on these two fractions as soon as they can be prepared on a sufficiently large scale.

#### Antibiotics from Cabbage

Cabbage has been found to be a source of antibiotics. A crystalline antifungal substance has been isolated, in very small amounts, from a methanolic extract of cabbage. Two ether active fractions have been separated from the supernatant solution. One of these fractions is active toward Gram-positive bacteria (S. aureus) and fungi, whereas, the other fraction is, in addition, active toward Gram-negative bacteria (E. coli) and acid-fast bacteria (Mycobacteria). Toxicological and in vivo antibiotic activity will be obtained, following further purification of the fractions.

#### Antibiotics from Bananas

The banana skin has been referred to as "Nature's bacteria proof wrapper". On investigation, the green banana skin and pulp were found to contain antifungal substances, whereas, ripe banana skin and pulp



(naturally and ethylene ripened) contained both antifungal and antibacterial substances. These results indicate that antibiotics in the banana skin and pulp appear during the ripening process. Of particular significance is the fact that an antibacterial factor active toward acid-fast bacteria (Mycobacteria) does not appear until the banana is well ripened, representing a color index 7 and 8. The antifungal substance, which inhibits the growth of disease-causing fungi, has been separated from the antibacterial fractions. Toxicological and in vivo antibiotic activity will be determined on these fractions when they are obtained in a purer state.

## ENZYMOLOGICAL INVESTIGATIONS

Enzyme Research Division, Albany, Calif.  
Arnold Kent Balls, Head

### Work on Citrus

Work has continued on phosphate transferase isolated from citrus fruit in an attempt to explain changes that take place in the ripening of the fruit, particularly reactions that change acid to sugar. Phosphatase studies on concentrated Valencia orange juice have shown that in freshly processed material the enzyme activity is mostly in the cloud, and after a year it was in the juice.

The study of poisoning of certain citrus enzymes having esterase activity by insecticidal substances related to di-isopropyl fulorophosphate has been extended, with particular attention to substances having possibilities as systemic plant insecticides.

Investigations of limonin (the bitter principle of citrus) have continued. The disappearance of limonin or its precursor from Navel oranges and from the pulp and peel of Valencia oranges during the course of ripening appears to be well established. Studies on the chemical structure of limonin have progressed, with particular attention given to degradation products in an effort to explain the bitter taste.

### Work on Cereals

Improvement in yields of crystals of barley malt alpha-amylase has been obtained by taking advantage of the great solubility of the enzyme in acetate buffer

at pH 4.7. Many requests for samples of the crystalline enzyme were met during the year. Studies were conducted on the nature of the hydrolysis catalyzed by malt amylase. Some of the physical properties of beta-amylase were determined, such as molecular weight (about 200,000) and density (1.35), and its mode of action on starch was studied. Beta-amylase in barley malt was also investigated.

Studies on the amylase produced by Bacillus macerans were pursued under a commercial fellowship. One achievement was a marked increase in yield of enzyme from cultures, largely through adjustment of pH with ammonia. Considerable progress was made on purification of the macerans amylase and on studies of the activity of purified samples. Schardinger dextrans, produced with macerans amylase, have been isolated and studied to some extent.



Factors that affect the preparation of an active lipase "cream" from castor beans received intensive study and it has been possible to produce a cream with reproducible activity.

Enzyme reactions in vegetables that have been scalded in preparation for freezing or dehydration have been given some attention. The reaction studied apparently decomposed the phospholipids. The enzyme in cabbage that decomposes soybean lecithin (a mixture of phospholipids) has been isolated.

#### RESEARCH & MARKETING ACT PROJECT

##### New and Improved Uses of Citrus Products

Two sulfhydryl sulfur compounds of citrus juice have been isolated and identified by chemical analysis. These compounds are the amino acid cysteine and the tripeptide glutathione. They account for at least 80 percent of the total of such compounds in citrus juice. Their enzymatic breakdown is now being studied.

LABORATORY OF FRUIT AND VEGETABLE CHEMISTRY

Pasadena, California

E. A. Beavens, In Charge

Fruit Products

A successful method for the canning and pasteurization of sucrose-type dates (Deglet Noor variety) developed during recent years is now in commercial use. Results with the invert-sugar type of dates (Khadrawi variety), which are moist and tender, indicate that they also are preserved well by the method developed for the sucrose type of date. Essentially the method consists of packing in sealed cans and pasteurization in boiling water for 30 minutes or at 170°F. for 65 minutes.

Preliminary studies have indicated that Navel oranges can be used in frozen citrus purees (and in turn in frozen desserts and bakery goods) without evidence of bitter flavor. They are under severe handicap, however, because of the bitter flavor in processed Navel juice. Results of recent research have indicated that tomato pectic-acid-depolymerase added to the juice will remove or prevent a large part of the bitterness and also prevents gelling of frozen purees. Commercial enzyme preparations (such as Pectinol) and activated carbon offer some possibility for the control of the bitterness in Navel orange products. Work is under way on chemical changes in citrus purees in freezing storage and on the bitter principle in Navel orange juice and methods for control.

Vegetable Products

Studies on commercial and laboratory-prepared tomato pastes have revealed that preheating temperatures commonly used are insufficient for the inactivation of pectic enzymes. Resulting losses of pectin during processing have sometimes lowered the consistency of the pastes beyond the point of desirability. Concentration to a solids content higher than the standard 25 percent, as sometimes practiced, is costly and also is a relatively ineffective way to increase consistency. Recommendations are now available that make possible the production of high-quality pastes of legal solids content. Further studies will be made on other factors that may contribute to consistency of pastes.

RESEARCH AND MARKETING ACT PROJECTS

New Laboratory at Pasadena, Calif.

The new building constructed for the Fruit and Vegetable Chemistry Laboratory in Pasadena with RMA funds was completed approximately on schedule. The building was dedicated Apr. 14, 1949, with appropriate ceremony. Numerous visitors have inspected the building for the purpose of obtaining suggestions for the construction of other research buildings.



New and Improved Uses of Citrus Products

Studies have been completed on the water-soluble volatile flavoring constituents of fresh and canned orange and grapefruit juices. Eight compounds were isolated, including alcohols, aldehydes, esters, and ketones. Work has continued on chromatographic separation of volatile oils isolated from the juices. New work under this project involves improvements in quality of frozen citrus concentrates. Some work is being done on effects of storage temperatures on the quality of concentrated orange juice, including chemical changes. Another new phase of research is concerned with nitrogenous constituents of citrus juices, particularly the amino acids. Orange juice, for example, may contain sufficient amounts of amino acids to be of nutritional significance for young children.

## FRUIT AND VEGETABLE PRODUCTS LABORATORY

Pullman, Washington

A. M. Neubert, In Charge

### Fruit Products

Studies on application of biologically active compounds to Perfection apricots and Italian prunes to determine whether such dipping treatments might soften the skins and yield more attractive products gave unpromising results. Skins of prunes were not softened; those of the apricots were softened but the flesh beneath became too soft for canning. Methods for the production of apricot nectar from the Wenatchee Moorpark variety, developed earlier, are now being used commercially. Results of 12 years of study on processing of freestone peaches have been widely used in recent years.

### Vegetable Products

Results of comparative studies on measurements of maturity in sweet corn have been summarized as follows: Refractive index, found to be one of the most rapid, accurate, and practical methods; crude starch and alcohol-insoluble solids content, accurate but time-consuming; rapid colorimetric method for starch, unreliable results because of seasonal variation in type of starch; crude carotene content, reliability indicated in preliminary tests; age and sugar content, poor results; Brown-Duvel moisture method, borderline in accuracy.

Studies were begun on the canning and freezing quality of 13 varieties of green beans in Idaho. Preliminary results were obtained, and these studies will continue.

Results of a three-year study recently completed have provided canners with information on tomato varieties and processing methods most suitable for high quality in the Northwest.

## RESEARCH AND MARKETING ACT PROJECT

### Recovery of Edible Juice from Pear Waste

A process was developed for the economical recovery of soluble solids from pear canning waste. It involves conversion of milled waste into a calcium pectinate gel which can be easily pressed to give high yields of clear juice. The juice is purified to a colorless liquid suitable for use as a sweetening ingredient in canned pears by treatment with an ion exchange resin and decolorizing carbon. The practicability of the lime gelation process was demonstrated on a commercial scale and the juice purification process on a pilot plant scale. The commercial and pilot plant studies were made at the Apple Growers Association cannery, Hood River, Oregon.



MICROBIOLOGY RESEARCH DIVISION  
Located at  
Agricultural Research Center  
Beltsville, Maryland

Mathilde Solowey, Acting Head of Division

MICROBIOLOGY OF DRIED EGGS AND RELATED EGG PRODUCTS

Development of an Improved Method for the Fermentation of  
Egg White

A method for the rapid fermentation of egg white by the use of non-proliferating cell suspensions of a streptococcal strain has been developed on a laboratory scale. It has been demonstrated that several different microorganisms are able to produce the desired results, although further and more detailed comparative studies are in progress. By the method employed, fermentation of egg white (utilization of the carbohydrate) can take place within 4 hours, with concomitant drop in pH to the desired level. During the fermentation, the growth of contaminants that usually produce objectionable flavors and odors in egg white is inhibited. The dried fermented egg white thus produced yields a product that is quite satisfactory according to the usual chemical, physical, and organoleptic criteria. Optimum conditions for fermentation, as well as adaptability of the process to pilot plant and industrial operation, are now being studied.

The Antibiotic Activity of Polymyxin and Other Anti-Bacterial  
Agents Against Salmonella Organisms Isolated from Dried Egg  
and Related Egg Products

The mode of action, minimal inhibitory dose, effect of size of inoculum and development of resistance to aureomycin, chloromycetin, and polymyxin B has been studied in vitro for 3 Salmonella species isolated from dried egg powder. All three antibiotics appear to be equally effective against the three test strains studied. Aureomycin and chloromycetin were found to be bacteriostatic in the concentrations employed whereas polymyxin B was found to be bactericidal. The effectiveness of aureomycin was not altered by the size of the inoculum, whereas polymyxin B and chloromycetin were so influenced. No significant development of resistance to any of the antibiotics by the organisms tested was observed. In vivo testing, using the developing

chick embryo, has been initiated, and the minimal lethal dose has been determined for the strains studied. Further studies to evaluate the in vivo effectivity of antibiotics for Salmonella strains isolated from egg powder are being conducted on a cooperative basis with the National Institutes of Health.

RMA PROJECT #401: PREPARATION OF PRE-COOKED FROZEN FOODS, AND THE POSSIBLE OCCURRENCE AND SIGNIFICANCE OF PATHOGENIC ORGANISMS IN FROZEN FOODS

Occurrence and Significance of Salmonella Organisms in Commercially Frozen Foods

A bacteriologist recently assigned to this project has devoted primary attention to developing methods of sampling frozen foods and to evaluating the relationship of different conditions inherent to the sampling procedure. Samples of frozen food products representing chicken a la king, chicken chow mein and vegetable chow mein have been examined for viable cell count, gram negative bacteria count and for the presence or absence of Salmonella. One of twenty samples thus far studied has shown contamination with Salmonella. Study of the general microbial flora indicate the presence of a wide variety of microorganisms.

RMA PROJECT #440: IMPROVEMENT OF MICROBIAL QUALITY OF EGGS, EGG PRODUCTS, POULTRY AND POULTRY PRODUCTS

Improvement of Microbiological Methods for the Detection of Salmonella Organisms in Dried Eggs and Related Egg Products

Continued investigations have been made on the comparison and evaluation of different methods of isolating Salmonella from dried egg powder and related egg products. Several synthetic media of known composition are being evaluated with satisfactory results on the samples thus far studied. Specifically it appears that a lithium chloride-asparagine broth in some respects is comparable to Selenite-F as an enrichment medium for the Salmonella.

In studying the role of certain substances as nutritive requirements for the Salmonella it has been shown that among others, DL-alpha-alanine, asparagine, sodium citrate, glycerol, lactic acid, and succinic acid are utilizable as energy sources for growth.



Investigation of the Pathogenicity for Humans of Salmonella.  
Organisms Isolated from Dried Egg and Related Egg Products

The contractual phase of this project has progressed to the following extent: strains of three Salmonella species have been tested for sulfonimide, streptomycin, and chloromycetin sensitivities, infectivity for animals prior to feeding to humans. Fifty to seventy-five human volunteers have been checked for use in these tests by studying their blood and stool cultures prior to test feeding. Three strains of each of the three type strains have been fed in graded doses to volunteers in groups of 6, and the course of activity studied.

NAVAL STORES RESEARCH DIVISION

E. L. Patton, Head

Approximately two-thirds of the research of this Division is carried out at New Orleans; the remainder at the Naval Stores Station, Olustee, Florida.

NAVAL STORES PRODUCTION, PROCESSES, AND  
EQUIPMENT INVESTIGATIONS

Selection of Cups for Acid-Stimulated Gum

Testing of present and possible future turpentine cup materials under conditions of acid stimulation was continued. Commercial costing materials were damaged by turpentine in less than one year, but metals and a special cement mixture were little affected. At the end of 1949 the testing program was reduced to those materials which showed promise of commercial use at reasonable price levels.

Improved Processing Methods and Equipment

A packed column continuous still for the distillation of turpentine and rosin was built in commercial size and given preliminary tests. It replaces the 12 inch continuous still, which could not reach the large commercial capacity requirements.

An entrainment separating column was developed for use with present commercial gum stills to meet new requirements by consumers for low-acid turpentine. Several packing materials were tested, although so far the use of the empty column is preferable.

A new type of filter was tested for use in the Government-patented Olustee Process of gum cleaning, under which most of the processing industry operates. The filter which is self-cleaning eliminates the necessity of daily cleaning by hand, and will be recommended to the processing industry.

Help When Needed

Technical assistance by the staff of the Naval Stores Station in solving production and control problems in gum processing plants was continued. Most problems involved the lowering of turpentine acidity and the prevention of haze in rosin. Reduction of turpentine acidity was accomplished by the use of the velocity reduction column developed this year and the prevention of haze in rosin was met through more rigid adherence to the recommendations for gum washing, previously made by the Naval Stores Station.



## INVESTIGATIONS OF THE COMPOSITION, PROPERTIES COMPONENTS AND DERIVATIVES OF NAVAL STORES

### Composition of Acid-Stimulated Pine Gum

Determination of the composition of the pine gum obtained by acid stimulation and by fungus stimulation which showed that such methods of stimulation do not significantly affect the composition of the turpentine and rosin produced from such pine gum has facilitated commercial introduction of acid stimulation which is now being generally adopted by producers of pine gum.

### New Compounds from Turpentine

Many new compounds have been obtained from hydrocarbons obtainable from turpentine. These include oxygenated compounds such as acids, ketones, and peroxides, as well as halogenated compounds. Aside from theoretical interest in such new compounds, some of these should have practical industrial utility; for example, as catalysts, oil additives, and plasticizers. Certain peroxides from hydrocarbons obtainable from turpentine have been found to be highly effective catalysts for the production of synthetic rubber at low temperatures ("cold rubber"). For example, the peroxide from pinane is a more effective catalyst than cumene hydroperoxide which is now being used commercially. The general applicability of a new type of reaction to produce highly halogenated compounds from terpenes obtainable from turpentine has been established and a number of products such as the addition of  $\beta$ -pinene with carbon tetrachloride and the addition product of p-pinene with hexachloroethane were obtained using this reaction. Such products are potentially useful as insecticides, lubricating oil additives, flameproofing agents, and plasticizers.

## INVESTIGATION OF USES, HANDLING, AND TRANSPORTATION OF NAVAL STORES

### New Type of Metal Resinate Developed

A new method has been developed for the preparation of metal resinates which gives products of higher metal content and better solubility in hydrocarbon solvents than those prepared by any previously reported method. This development of a new type of metallic resinate having both a higher metal content, lower acid number, and better solubility in hydrocarbon solvents should enable the naval stores industry to regain a considerable portion of the market for resinate driers that has been lost to the naphthenate driers. It should also permit the inclusion of calcium, zinc, and magnesium resinates in a much wider variety of varnish formulations than has been possible with the present type of metallic resinates. The process developed in this laboratory is the only method known by which neutral metallic resinates that are permanently refusible can be prepared.

RESEARCH AND MARKETING ACT INVESTIGATIONS

Pilot-Plant Production of Maleo-Pimaric Acid  
from Gum Rosin (RM:a-100)

A laboratory method for the production of the maleic anhydride adduct of levopimaric acid has been successfully applied on a pilot-plant scale at the Field Station of the Naval Stores Division at Olustee, Florida. For the convenience of manufacturers and consumers the trade name "Maleo-Pimaric Acid" was adopted for the substance.

It is the first chemical product made directly from crude pine gum without wasteful intermediate processing and transportation between plants. It also offers the first possibility of establishing a chemical industry in the processing of gum naval stores, which to date has been under the disadvantage of selling its products (turpentine and rosin) on a fluctuating commodity market to a chemical industry which requires stable prices for most efficient operation and economy. A number of production difficulties have been overcome, and samples of the latest high quality have been submitted to 93 individuals and companies in the chemical and pharmaceutical industries for commercial evaluation at their request.



PHARMACOLOGICAL INVESTIGATIONS  
Pharmacology Division, Albany, Calif.  
Floyd DeEds, Head

Rutin, quercetin, quercitrin, hesperidin, and grape phlobotannin have been shown to exert a protective action against experimental frostbite in rabbits. Chronic toxicity studies on quercetin and quercitrin have been completed except for histopathological examination of tissues. Hesperidin chalcone and quercitrin have been shown to have the same effect on cutaneous capillaries as rutin.

Chronic toxicity studies on subtilin have been completed except for histopathological examination of tissues of the experimental animals. If the examination of the tissues of animals show no evidence of injury, this evidence of non-toxicity together with the evidence based on growth, appearance and behavior of the animals will establish the non-toxicity of orally ingested subtilin.

The trypsin inhibitor of raw white beans inhibited the growth of albino rats and produced hypertrophy of the pancreas. Raw black-eyed beans having a comparable amount of trypsin inhibitor did not produce these toxic reactions.

Feeding of xylose to albino rats has been shown to inhibit growth and produce cataract when the xylose constituted 15 per cent or more of the diet. Thus use of xylose by diabetic persons is contra-indicated.

Paired-feeding experiments with albino rats has shown that the inhibition of growth produced on a diet containing known amounts of cottonseed pigment glands is due to a specific toxic action over and above the inhibition due to decreased food intake. This emphasizes the desirability of removing all pigment gland material from cottonseed meal.

Investigations have been completed on acute and chronic toxicity of four inhibitors of biological activity in seeds, namely vinyl propionate, ethylene chlorohydrin, propylene glycol dipropionate, and 1,3-dimethyl-4-6-bis(chloromethyl)benzene.

Chronic toxicity studies on 2-amino-pyridine proposed for use in the prevention of stem-end rot in oranges have been completed, except for the histopathological examination of the animal tissues. If this examination confirms this low toxicity, the compound may prove of great economic value in reducing the loss of Florida oranges due to stem-end rot.

Chronic toxicity studies (except for histopathological examination of tissues) have been completed on itaconic acid and 4-chlorophenoxyacetic acid. Studies have been started on hydroquinone derivatives proposed as antioxidants for the carotene of alfalfa, on metal pectinates, on mercury treated casein fibers, and the spray residues present in pear waste pomace.



NATURAL RUBBER EXTRACTION & PROCESSING INVESTIGATIONS  
U. S. Natural Rubber Research Station  
Salinas, California  
I. C. Feustel, In Charge

The present program of extraction and processing investigations concerned with natural rubber was initiated August 1, 1947. In this program the Bureau of Agricultural and Industrial Chemistry collaborates with the Bureau of Plant Industry, Soils and Agricultural Engineering at the U. S. Natural Rubber Research Station at Salinas, Calif. On July 1, 1949, curtailment of operations went into effect to meet a reduction of approximately 40 percent in appropriations made by Congress for the fiscal year 1950. Investigations on latex extraction and on byproduct utilization have accordingly been discontinued and other work has been reduced materially in scope.

Milling of Lush Shrub

Confirming evidence of the superiority of the processing of lush or freshly harvested shrub (instead of shrub that has been conditioned by field exposure and storage) has been obtained in a direct comparison of the two methods. The new process effects complete coagulation of latex by thorough crushing and hammer milling, regardless of moisture content, and eliminates storage and drying. It also results in higher yields, greater uniformity, and higher quality of rubber. This development provides a basis for studies on continuous processing, which will soon be undertaken.

Deresination

Continued work on deresination by acetone extraction of guayule shrub prior to milling has confirmed earlier evidence that this method produces rubber of substantially improved quality, containing low percentages (1 to 2 percent) of resin. The acetone is recoverable, and it seems probable that this step can be made part of a continuous operation. Samples of the deresinated rubber have received favorable comment from industrial concerns. Promising results have also been obtained from a new method for extracting resins directly from the wet rubber "worms" which are formed at one stage of the shrub milling process.

Resin Characterization

Guayule resin has been found to be separable into three fractions, based on solubility, as follows: (1) water solubles (betaine, levulins, various inorganic salts), (2) aliphatic hydrocarbon (hexane) solubles (waxes, low-grade "rubber", essential oils, terpene-like solids including parthenyl cinnamate), and (3) aqueous alcohol solubles (mostly high-molecular-weight gum-like resin). Methods for separation of parthenyl cinnamate and essential oils.



and also for betaine, are subjects of current public-service patent applications.

#### Low-Temperature Testing of Guayule Rubber

Samples of latex rubber, deresinated rubber, and resinous rubber are being tested for serviceability at low temperatures (-70°F. or lower) by various government agencies. The first results (obtained by Government Rubber Evaluation Laboratories in Akron, Ohio) indicate that the samples had no significant advantages for low-temperature applications.

#### Aging Studies

Aging studies on crude and vulcanized guayule rubber (both resinous and deresinated) are being undertaken. Apparatus for oxygen-absorption investigations has been completed. This work will be concerned with antioxidants, oxidative effects of metals, resins, resin fractions, and various processing treatments; it is expected to indicate improved measures for the stabilization of guayule rubber.



